WRINKLE REDUCING COMPOSITION

10

5

15

Field of the invention

20

The present invention relates to fabric care compositions and to a method for treating fabrics in order to improve various properties of fabrics, in particular, reduction or removal of unwanted wrinkles.

25

Background of the invention

30

35

Wrinkles in fabrics are caused by the bending and creasing of the textile material which places an external portion of a filament in a yarn under tension while the internal portion of that filament in the yarn is placed under compression. Particularly with cotton fabrics, the hydrogen bonding that occurs between the cellulose molecules contributes to keeping wrinkles in place. The wrinkling of fabric, in particular clothing, is therefore subject to the inherent tensional elastic

deformation and recovery properties of the fibers which constitute the yarn and fabrics.

In the modern world, with the increase of hustle and bustle and travel, there is a demand for a quick fix which will help to diminish the labor involved in home laundering and/or the cost and time involved in dry cleaning or commercial laundering. This has brought additional pressure to bear on textile technologists to produce a product that will sufficiently reduce wrinkles in fabrics, especially clothing, and to produce a good appearance through a simple, convenient application of a product.

It has now been found that the use of specific adhesive polymers fulfills such a need. Specific polymers are polymers which are used in hair care such as described in EP-A-412704, WO 97/34572 and US 5,120,532.

15

20

25

10

5

Accordingly, the present invention reduces wrinkles from fabrics, including clothing, dry cleanables, and draperies, with a reduced need for ironing. The present invention can be used on damp or dry clothing to relax wrinkles and give clothes a ready to wear look that is demanded by today's fast paced world. The present invention also essentially eliminates the need for touch up ironing usually associated with closet, drawer, and suitcase storage of garments.

When ironing is desired however, the present invention can also act as an excellent ironing aid. The present invention makes the task of ironing easier and faster by creating less iron drag. When used as an ironing aid, the composition of the present invention produces a crisp, smooth appearance.

An additional benefit of the composition of the present invention is an improved garment shape, body and crispness.

30

A further additional benefit to invention composition is the variety of fabric that can be treated from the more resistant to the more delicate including fabric made of cotton, polycotton, polyester, viscose, rayon, silk, wool, etc...

It is also an object of the invention to provide solubilised compositions comprising the co-polymer. Traditionally, the co-polymer is solubilised with high levels of

20

25

30

organic solvents. However, such high levels often give rise to environmental and safety concerns. Thus, it is another object of the invention to provide solubilised wrinkle reducing compositions which are environmentally compatible.

5 It has now been found that water-soluble surfactants are desirable solubilising agents.

Summary of the invention

The present invention relates to a wrinkle reducing composition, comprising:

A-a wrinkle reducing active, comprising an adhesive polymer selected from:

I-a silicone grafted adhesive copolymer having a molecular weight of from 10,000 to 1,000,000, which has an organic polymeric backbone having grafted to it monovalent siloxane polymeric moieties, said copolymer comprising C monomers and components selected from the group consisting of A monomers, B monomers, and mixtures thereof; wherein:

- a. A is at least one free radically polymerizable organic monomer, the amount by weight of A monomer, when used, being up to about 100% of the total weight of all monomers in said backbone;
- b. B is at least one reinforcing monomer copolymerizable with A, the amount by weight of B monomer, when used, being up to about 100% of the total weight of all monomers in said backbone, said B monomer being selected from the group consisting of polar monomers and macromers; and

c. C is a polymeric monomer copolymerizable with said A monomer and said B monomer, said C monomer having a having a molecular weight of from about 1,000 to about 50,000; and having the general formula:

 $X(Y)_n Si(R)_{3-m} (Z)_m$

5 wherein:

10

20

25

30

35

X is a vinyl group copolymerizable with said A monomers and said B monomers;

Y is a divalent linking group;

R is a hydrogen, lower alkyl, aryl or alkoxy;

Z is a monovalent siloxane polymeric moiety having a number average molecular weight of at least about 500, is essentially unreactive under copolymerization conditions, and is pendant from said polymeric backbone after copolymerization;

n is 0 or 1;

m is an integer from 1 to 3; and wherein C comprises from about 0.01% to about 50% of the copolymer;

II-a polymer having a weight average molecular weight from 5000 to 1,000,000, which has an organic polymeric backbone, said copolymer comprising monomers selected from the group consisting of A monomers, B monomers and mixtures thereof, wherein said copolymer is prepared by the polymerisation combination of the following relative weight percentages of said A monomers, and said B monomers:

- a- from 0% to about 100%, by weight of said copolymer, of a hydrophobic, A monomer, free radically copolymerizable with said B monomers;
- b. from 0% to about 100%, by weight of said copolymer, of a hydrophilic reinforcing B monomer, copolymerizable with said A monomer, said B monomer being selected from the group consisting of polar monomers and macromers and mixtures thereof; and
- III- an adhesive block copolymer having a weight average molecular weight from about 10,000 grams/mole to about 10,000,000 grams/mole and which is formed from the free radical polymerization of an ethylenically unsaturated monomer with a silicone macroinitiator selected from the group consisting of

and combinations thereof, wherein each R is independently selected from the group consisting of C1-C10 alkyl, phenyl, C1-C10 alkyl-substituted phenyl, and mixtures thereof, X is a divalent radical, Y is selected from the group consisting of

and combinations thereof; and m, n, and p are positive integers independently having a value of 1 or greater; and

wherein the silicone macroinitiator has a number average molecular weight from about 500 grams/mole to about 500,000 grams/mole, and the ethylenically unsaturated monomers are copolymerizable and form one or more polymeric blocks within the adhesive block copolymer having a Tg value of from about -20°C to about 60°C.

IV-mixtures thereof;

B-a water-solubilising agent;

C-a liquid aqueous carrier, and

wherein when the polymer is as defined under (I) or (II) said water-solubilising agent is in amount of less than 5% by weight of the composition and is selected from water-soluble anionic surfactant, water-soluble nonionic surfactant, and mixtures thereof.

10

15

In another aspect of the invention, there is provided a method of reducing the wrinkles on fabrics which comprises the steps of contacting the fabrics with a composition of the invention.

In another aspect of the invention, there is also provided a process of solubilising the polymer by means of a water-soluble surfactant.

Still in a further aspect of the invention, there is provided a packaged composition comprising the composition of the invention in a spray dispenser.

10

Detailed description of the invention

15

20

25

30

35

A. Wrinkle reducing actives

The wrinkle reducing active for use in the present invention is an adhesive polymer. This adhesive polymer is selected from a silicone grafted adhesive polymer, an adhesive polymer comprising monomers selected from the group consisting of A monomers, B monomers and mixtures thereof, a silicone containing adhesive block polymer, and mixtures thereof.

By "adhesive" it is meant that when applied as a solution or dispersion to a surface and dried, e.g., the fabric fibers, the polymer forms films or welds onto the applied surface. Such a film or weld will have adhesive and cohesive strength, as is understood by those skilled in the art.

The term "hydrophobic" is used herein consistent with its standard meaning of lacking affinity for water, whereas "hydrophilic" is used herein consistent with its standard meaning of having affinity for water.

As used herein in relation to monomer units and polymeric materials, including the copolymers, "hydrophobic" means substantially water insoluble; "hydrophilic" means substantially water soluble. In this regard, "substantially water insoluble" shall refer to a material that is not soluble in distilled (or equivalent) water, at 25.degree. C., at a concentration of 0.2% by weight, and preferably not soluble at

15

35

0.1% by weight (calculated on a water plus monomer or polymer weight basis). "Substantially water soluble" shall refer to a material that is soluble in distilled (or equivalent) water, at 25.degree. C., at a concentration of 0.2% by weight, and are preferably soluble at 1.0% by weight. The terms "soluble", "solubility" and the like, for purposes hereof, corresponds to the maximum concentration of monomer or polymer, as applicable, that can dissolve in water or other solvents to form a homogeneous solution, as is well understood to those skilled in the art.

The polymers should have a weight average molecular weight of from about 10,000 to about 1,000,000 (preferably from about 30,000 to about 300,000).

Preferably, the backbone of the polymer will have a Tm above room temperature, and a Tm for the C monomeric unit below 0°C. As used herein, the abbreviation "Tg" refers to the glass transition temperature of the backbone of the polymer, and the abbreviation "Tm" refers to the crystalline melting point of the backbone, if such a transition exists for a given polymer.

I-Silicone grafted adhesive polymer

The hydrophobic polymers of the present invention can be described as graft copolymers. The term "graft copolymers" is familiar to one of ordinary skill in polymer science and is used herein to describe the copolymers which result by adding or "grafting" a polymeric chemical moiety (i.e. "grafts") onto another polymeric moiety commonly referred to as the "backbone". The backbone typically has a higher molecular weight than the grafts. Thus, graft copolymers can be described as polymers having pendant polymeric side chains, and as being formed from the "grafting" or incorporation of polymeric side chains onto or into a polymer. The polymer to which the grafts are incorporated can be homopolymers or copolymers, e.g. linear random or block polymers. Such polymers are derived from a variety of monomer units.

The silicone grafted adhesive copolymers of the copolymer component of the present invention comprise an organic backbone preferably a carbon backbone derived from ethylenically unsaturated monomers, such as a vinyl polymeric backbone, and a polysiloxane macromer grafted to the backbone. These copolymers are comprised of non-silicone containing, hydrophobic, free radically

polymerizable "A" monomers, copolymerizable with the "B" and/or "C" monomers; grafted polysiloxane-containing vinyl "C" monomers, copolymerizable with "A" and "B" monomers; and, optionally, non-silicone containing, hydrophilic reinforcing "B" monomers which are polymerizable with the "A" and/or "C" monomers.

The silicone grafted copolymers are characterized by polysiloxane moieties covalently bonded to and pendant from a polymeric carbon-based backbone. The backbone will preferably be a carbon chain derived from polymerization of ethylenically unsaturated monomers to which polysiloxane moieties are pendant.

The term "ethylenically unsaturated" is used herein to mean a material that contains at least one polymerizable carbon-carbon double bond, which can be mono-, di-, tri, or tetra-substituted. The backbone can also include ether groups, ester groups, amide groups, urethane groups and the like. The polysiloxane moieties can be grafted onto a copolymer backbone or the copolymer can be made by copolymerization of polysiloxane-containing polymerizable monomers at the chain end (e.g., ethylenically unsaturated monomers) with non-polysiloxane-containing copolymerizable monomers.

20

25

5

10

15

The term "copolymerizable" is used herein to describe a material which can be reacted with another material (e.g. the A monomers, B monomers and C monomers of the present invention) in a polymerization reaction using one or more conventional synthetic techniques, such as ionic, emulsion, dispersion, Ziegler-Natta, free radical, group transfer or step growth polymerization. The preferred copolymerization synthetic technique is free radical polymerization.

The polymers should have a weight average molecular weight of from about 10,000 to about 1,000,000, preferably from about 30,000 to about 300,000.

30

The silicone grafted polymers of the compositions of the present invention comprise "silicone-containing" (or "polysiloxane-containing") monomers, which form the silicone graft pendant from the backbone, and non-silicone-containing monomers, which form the organic backbone of the polymer.

35

The silicone grafted polymers should satisfy the following three criteria:

- (1) when dried the copolymer phase-separates into a discontinuous phase which includes the silicone portion and a continuous phase which includes the non-silicone portion;
- (2) the silicone portion is covalently attached to the non-silicone portion;
- 5 (3) the molecular weight of the silicone portion is from about 1,000 to about 50,000.

When used in a composition, such as a wrinkle reducing composition, the non-polysiloxane portion should permit the copolymer to deposit on the fabric.

10

15

20

The most preferred polymers comprise grafted to the backbone, a polydimethylsiloxane macromer having a weight average molecular weight of from about 1,000 to about 50,000, preferably from about 5,000 to about 40,000, most preferably about 15,000. The polymer is such that when it is formulated into the finished composition, when dried, the polymer phase separates into a discontinuous phase which includes the polydimethylsiloxane macromer and a continuous phase which includes the backbone.

Without being limited by theory, it is believed that the phase separation property provides a specific orientation of the copolymer which results in the desired combination of tactile feel, film- forming or adhesive benefits, and the ability to dry quickly and completely.

The phase-separating nature of the compositions of the present invention may be determined as follows:

The polymer is cast as a solid film out of a solvent (i.e., a solvent which dissolves both the backbone and the silicone). This film is then sectioned and examined by transmission electron micrography. Microphase separation is demonstrated by the observation of inclusions in the continuous phase. These inclusions should have the proper size to match the size of the silicone chain (typically a few hundred nm or less) and the proper density to match the amount of silicone present. This behavior is well documented in the literature for polymers with this structure (see, for example, S. D. Smith, Ph.D. Thesis, University of Virginia, 1987, and references cited therein).

30

10

15

20

25

30

35

A second method for determining phase-separating characteristics involves examining the enrichment of the concentration of silicone at the surface of a polymer film relative to the concentration in the bulk polymer. Since the silicone prefers the low energy air interface, it preferentially orients on the polymer surface. This produces a surface which is entirely covered by silicone even when the concentration of the silicone by weight in the whole polymer is low (2% to 20%). This is demonstrated experimentally by ESCA (electron spectroscopy for chemical analysis) of the dried film surface. Such an analysis shows a high level of silicone and a greatly reduced level of backbone polymer when the film surface is analyzed. (Surface here means the first few tens of Angstroms of film thickness.) By varying the angle of the interrogating beam the surface can be analyzed to varying depths.

These copolymers are comprised of monomers A, C and, optionally, B, which are defined as follows. A is at least one free radically polymerizable monomer or monomers. B, when used, comprises at least one reinforcing monomer copolymerizable with A and is selected from the group consisting of polar monomers and macromers. When used, B may be up to about 100%, preferably up to about 80%, more preferably up to about 20%, of the total monomers in the backbone. Monomer C comprises from about 0.01% to about 50.0% of the total monomers in the copolymer.

The particular relative amounts of A monomers, B monomers, and C monomers can vary as long as the polymer backbone is soluble or dispersible in the water-soluble solubilising agent hereof and the silicone grafted copolymer exhibits phase separation when dried.

The polymers are prepared by the polymerization combination of A monomer, C monomer and, if used, B monomer. The copolymer composition is characterized by the amount of each monomer charged to the polymerization reaction vessel. In general, the silicone grafted, adhesive copolymer will comprise from about from 0% to about 100% (preferably from about 5% to about 98%, more preferably from about 30% to about 90%) of monomer A of the total monomers in the backbone, from about 0% to about 100% (preferably from about 7.5% to about 80%) of monomer B of the total monomers in the backbone, and from about 0.01% to about 50% (preferably from about 0.5% to about 40%, most

preferably from about 2% to about 25%) of monomer C of the total monomers in the polymer.

The combination of the A and B monomers preferably comprises from about 50.0% to about 99.9% (more preferably about 60% to about 99%, most preferably from about 75% to about 95%) of the polymer. The composition of any particular copolymer will help determine its formulational properties. In fact, by appropriate selection and combination of particular A, B and C components, the copolymer can be optimized for inclusion in specific vehicles.

10

25

30

35

5

For example, polymers which are soluble in an aqueous formulation preferably have the composition: from about 0% to about 90% (preferably from about 5% to about 70%) monomer A, from about 10% to about 98% (preferably from about 10% to about 80%) monomer B, and from about 1% to about 40% monomer C.

Polymers which are dispersible have the preferred composition: from about 0% to about 90% (more preferably from about 5% to about 80%) monomer A, from about 10% to about 80% (more preferably from about 10% to about 60%) monomer B, and from about 1% to about 40% monomer C.

More preferably, the polymers comprise from about 5% to about 98% A monomer, from about 0.01% to about 50% C monomer, and from 0% to about 98% B monomer.

The hydrophobic organic A monomer, which is free radically copolymerizable with B monomers and C monomers, is selected from the group consisting of acrylic acid esters, methacrylic acid esters, vinyl compounds, vinylidene compounds, unsaturated hydrocarbons, C₁ -C₁₈ alcohol esters of organic acids and organic acid anhydrides, and mixtures thereof. Representative examples of hydrophobic A monomers are acrylic or methacrylic acid esters of C₁ -C₁₈ alcohols, such as methanol, ethanol, methoxy ethanol, 1-propanol, 2-propanol, 1-butanol, 2-methyl-1-propanol, 1-pentanol, 2-pentanol, 2-methyl-1-butanol, 1-methyl-1-butanol, 3-methyl-1-butanol, 1-methyl-1-pentanol, 2-methyl-1-pentanol, 3-methyl-1-butanol, 3-heptanol, benzyl alcohol, 2-octanol, 6-methyl-1-heptanol, 2-ethyl-1-butanol, 3,5-dimethyl-1-hexanol, 3,5,5-tri methyl-1-hexanol, 1-dodecanol, 1-hexadecanol, 1-octa decanol, and the like, the

WO 00/08249 PCT/US98/16127

alcohols having from about 1-18 carbon atoms with the number of carbon atoms preferably being from about 1-12; styrene; polystyrene monomer; vinyl acetate; vinyl chloride; vinylidene chloride; vinyl propionate; alpha-methylstyrene; t-butylstyrene; butadiene; cyclohexadiene; ethylene; propylene; vinyl toluene; t-butyl acrylate, t-butyl methacrylate, n-butyl methacrylate, n-butyl acrylate, isobutyl methacrylate, 2-ethylhexyl methacrylate, methyl methacrylate, and mixtures thereof.

Other hydrophobic organic A monomer include the condensation polymer such as the polyamides, polyesters, polyurethanes, and mixtures thereof.

Preferred A monomers are vinyl monomers, more preferably selected from t-butyl acrylate, t-butyl methacrylate, t-butyl styrene, n-butyl methacrylate, n-butyl acrylate, isobutyl methacrylate, 2-ethylhexyl methacrylate, methyl methacrylate, and mixtures thereof.

In these polymers, it is more preferred that A be selected from t-butyl acrylate, t-butyl methacrylate, n-butyl methacrylate, n-butyl acrylate, and mixtures thereof, since such polymers can be dissolved directly in cyclomethicone solvents without requiring co-solvents.

The hydrophilic, reinforcing B monomer, which is copolymerizable with the A and C monomers, is selected from the group consisting of unsaturated organic monoand polycarboxylic acids. unsaturated (meth)acrylates, unsaturated (meth)acrylamides, unsaturated (meth)acrylate alcohols. unsaturated aminoalkylacrylates, unsaturated organic acid anhydrides, unsaturated esters of organic acid anhydrides, hydrophilic unsaturated vinyl compounds, hydrophilic unsaturated allyl compounds, hydrophilic unsaturated imides, salts of the foregoing compounds, and mixtures thereof.

30

35

5

15

20

25

Representative examples of hydrophilic reinforcing B monomers include acrylic acid, methacrylic acid, N,N-dimethylacrylamide, dimethyl aminoethyl methacrylate, quaternized dimethylaminoethyl methacrylate, methacrylamide, N-t-butyl acrylamide, t-butyl acrylamide, maleic acid, maleic anhydride and its half esters, crotonic acid, itaconic acid, acrylamide, acrylate alcohols, hydroxyethyl methacrylate, diallyldimethyl ammonium chloride, vinyl pyrrolidone, vinyl ethers

(such as methyl vinyl ether), maleimides, vinyl pyridine, vinyl imidazole, other polar vinyl heterocyclics, styrene sulfonate, allyl alcohol, vinyl alcohol (such as that produced by the hydrolysis of vinyl acetate after polymerization), salts of any acids and amines listed above, and mixtures thereof.

5

10

15

Other hydrophilic, reinforcing B monomer include the condensation polymer such as the polyisocyanates, polyalcohols, polyamines, polyethylene glycol and mixtures thereof. When a polyethylene imine is used, a dual benefit may be obtained: that of dewrinkling from the polymer and that of color care by the polyethyleneimine unit.

Preferred B monomers include monomers selected from the group consisting of acrylic acid, methacrylic acid, N,N-dimethyl acrylamide, methacrylamide, N t-butyl arylamide, dimethylamino ethyl methacrylate, t-butyl acrylamide, vinyl pyrrolidone, salts thereof and alkyl quaternized derivatives thereof, and mixtures thereof.

The polysiloxane-containing C monomer, copolymerizable with the A and B monomers, is exemplified by the general formula:

20 $X(Y)_n Si(R)_{3-m} Z_m$

wherein X is an ethylenically unsaturated group copolymerizable with the A and B monomers, such as a vinyl group;

Y is a divalent linking group;

R is a hydrogen, hydroxyl, lower alkyl (e.g. C_1 - C_4), aryl, alkaryl, alkoxy, or alkylamino;

Z is a monovalent siloxane polymeric moiety having a number average molecular weight of at least about 500, is essentially unreactive under copolymerization conditions, and is pendant from the polymeric backbone described above;

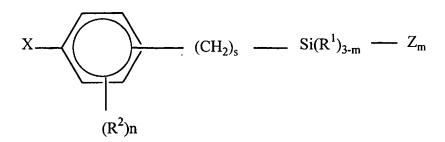
n is 0 or 1; and

m is an integer from 1 to 3.

C has a weight average molecular weight of from about 1,000 to about 50,000, preferably from about 5,000 to about 40,000, most preferably from about 10,000 to about 30,000.

35

Preferably, the C monomer has a formula selected from the following formulas:



or

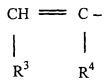
5

10

$$X \longrightarrow O \longrightarrow (CH_2)_q - (O)_p - Si(R^1)_{3-m} Zm$$

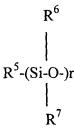
In these structures s is an integer from 0 to about 6, preferably 0, 1, or 2, more preferably 0 or 1; m is an integer from 1 to 3, preferably 1; p is 0 or 1; q is an integer from 2 to 6; R^2 is C_1 - C_{10} alkyl or C_7 - C_{10} alkylaryl, preferably C_1 - C_6 alkyl or C_1 - C_{10} alkylaryl, more preferably C_1 - C_2 alkyl; n is an integer from 0 to 4,

preferably 0 or 1, more preferably 0; R1 is hydrogen, hydroxyl, lower alkyl, alkoxy, alkylamino, aryl, or alkaryl, preferably R1 is alkyl; R" is alkyl or hydrogen; X is



5

R³ is hydrogen or —COOH preferably R³ is hydrogen; R⁴ is hydrogen, methyl or --CH2 COOH, preferably R4 is methyl; Z is



R⁵, R⁶, and R⁷, independently are lower alkyl, alkoxy, alkylamino, aryl, alkaryl, 10 hydrogen or hydroxyl, preferably R5, R6, and R7 are alkyls; and r is an integer of from about 5 to about 700, preferably about 60 to about 400, more preferably r is from about 80 to 200. Most preferably, R^5 , R^6 , and R^7 are methyl, p=0, and q=3.

15

The polymers of the present invention are prepared by the polymerization combination of A monomers, C monomers and, if used, B monomers. Disclosure of these polymers and preparation thereof are disclosed in EP412704, WO 97/34572, as well as WO 97/15275.

20

25

30

The silicone grafted copolymers can be synthesized by free radical polymerization of the polysiloxane-containing monomers with the nonpolysiloxane-containing monomers. The general principles of which are well understood. See, for example, Odian, "Principles of Polymerization", 3rd edition, John Wiley & Sons, 1991, pp. 198-334. The desired A, B, and C monomers are all placed in a reactor, along with a sufficient amount of a mutual solvent so that when the reaction is complete the viscosity of the reaction is reasonable. Typical monomer loadings are from about 20% to about 50%. Undesired terminators, especially oxygen, are removed as needed. This is done by evacuation or by purging with an inert gas, such as argon or nitrogen. The initiator is introduced and the reaction brought to the temperature needed for initiation to occur, assuming thermal initiators are used. Alternatively, redox or radiation initiation can be used. The polymerization is allowed to proceed as long as needed for a high level of conversion to be achieved, typically from a few hours to a few days. The solvent is then removed, usually by evaporation or by precipitating the copolymer by addition of a nonsolvent. The copolymer can be further purified, as desired.

By way of example, Polymers I, II and III, described above, are synthesized in the following manner. There are numerous variations on these procedures which are entirely up to the discretion of the synthetic chemist (e.g., choice of degassing method and gas, choice of initiator type, extent of conversion, reaction loading, etc). The choice of initiator and solvent are often determined by the requirements of the

particular monomers used, since different monomers have different solubilities and different reactivities to a specific initiator.

Polymer I: Place 10 parts acrylic acid, 70 parts n-butylmethacrylate, and 20 parts 20K PDMS macromer in a flask. Add sufficient ethyl acetate to produce a final monomer concentration of 40%. Add initiator, benzoyl peroxide, to a level of 0.5% by weight relative to the amount of monomer. Evacuate the vessel, and refill with nitrogen. Heat to 60.degree. C. and maintain this temperature for 48 hours while agitating. Terminate the reaction by cooling to room temperature, and dry off the ethyl acetate by pouring the reaction mixture into a teflon-coated pan and placing in a vacuum oven.

25

30

35

5

10

15

20

Polymer 11: Place 20 parts N,N-dimethylacrylamide, 60 parts isobutylmethacrylate, and 20 parts silicone macromer in a reaction vessel fitted with a temperature probe, reflux condenser, inlet port, and argon sparge. Add sufficient toluene to bring the final monomer concentration to 20% by weight. Sparge with argon for 1 to 2 hours. While sparging, heat to 62.degree. C. in a water bath. Add initiator, azobisisobutyronitrile, to a level of 0.25% by weight relative to the weight of monomer present. Maintain temperature at 62 degree. C., with a sufficient rate of argon flow to keep the solution mixed. Monitor the reaction visually, ensuring that no phase separation of reactants occurs during polymerization. If any turbidity is observed, add sufficient warm degassed toluene to eliminate the turbidity. Continue to monitor throughout the reaction. Terminate the reaction after 4 to 6 hours and purify as with Polymer I.

Polymer III: Place 10.5 parts N,N-dimethylmethacrylamide, 56 parts isobutyl methacrylate, 3.5 parts 2-ethylhexylmethacrylate, and 30 parts 10K PDMS macromer in a reaction vessel fitted with an argon sparge, temperature probe, reflux condenser and inlet port. Add sufficient toluene or isopropanol to bring the final monomer concentration to 20% by weight. Begin stirring and sparge with argon for 1 hour. While sparging, heat to 60.degree. C. in a water bath. Add initiator, azobisisobutyronitrile, to a level of 0.25% (if toluene is the solvent) or 0.125% (if isopropanol is the solvent) by weight relative to the weight of monomer present. Continue stirring and a slow argon sparge and maintain the reaction temperature at 60.degree. C. Allow to react for 6 hours. Terminate the reaction and remove the solvent as with Polymer II.

15

20

25

10

5

As an alternative to a batch reaction, the copolymer can be made by a semi-continuous or continuous process. In the semi-continuous process, two or more additions of monomers are made during the polymerization reaction. This is advantageous when the copolymer is made of several monomers which react during the polymerization at different rates. The proportions of monomers added to the reaction at the separate points of addition can be adjusted by one of ordinary skill in the art such that the polymers of the final product have a more uniform structure. In other words, the polymers of the final product will have a more consistent monomer content distribution for each of the monomer types charged to the reaction.

Examples of related copolymers and how they are made are described in detail in U.S. 4,693,935, US, 4,728,571, both of which are incorporated herein by reference.

30

35

Additional silicone grafted polymers are also disclosed in U.S. 5,480,634, Hayama et al, issued Oct. 2, 1996, 5,166,276, Hayama et al., issued Nov. 24, 1992, 5,061,481, issued Oct. 29, 1991, Suzuki et al., 5,106,609, Bolich et al., issued Apr. 21, 1992, 5,100,658, Bolich et al., issued Mar. 31, 1992, 5,100,657, Ansher-Jackson, et al., issued Mar. 31, 1992, 5,104,646, Bolich et al., issued

Apr. 14, 1992, and EP-A-0,751,162, all of which are incorporated by reference herein in their entirety.

Exemplary silicone grafted polymers for use in the present invention include the following, where the composition of the copolymer is given as weight percentage of each monomer used in the polymerization reaction used to prepare the copolymer.

- (i) acrylic acid/n-butylmethacrylate/(polydimethylsiloxane (PDMS) macromer 20,000 molecular weight) (10/70/20 w/w/w)
 - (ii) N,N-dimethylacrylamide/isobutyl methacrylate/(PDMS macromer -20,000 molecular weight) (20/60/20 w/w/w)
- 15 (iii) N,N-dimethylacrylamide/isobutyl methacrylate/2-ethylhexyl methacrylate/ (PDMS macromer -10,000 molecular weight) (10.5/56/3.5/30 w/w/w/w)
 - (iv) N,N-dimethylacrylamide/(PDMS macromer -20,000 molecular wt) (80/20 w/w)
 - (v) t-butylacrylate/t-butyl methacrylate/(PDMS macromer-10,000 molecular wt) (56/24/20 w/w/w)
 - (vi) t-butylacrylate/(PDMS macromer -10,000 molecular wt) (80/20 w/w)

20

25

- (vii) t-butylacrylate/N,N-dimethylacrylamide/(PDMS macromer 10,000 molecular weight) (70/10/20)
- (viii) t-butylacrylate/acrylic acid/(PDMS macromer -10,000 molecular weight) 30 (75/5/20)
 - (ix) t-butyl acrylate/acrylic acid/ (PDMS macromer-10,000 molecular weight) (63/20/17)
- 35 II-Adh sive polymer comprising monomers selected from the group consisting of A monom rs, B monomers and mixtures th r of.

10

30

35

The parent polymer of the above defined silicone grafted polymer is also an adhesive polymer of use herein.

The polymer can be homopolymers or copolymers of hydrophobic and/or hydrophilic monomers, e.g. linear random or block polymers. Such polymers are derived from a variety of monomer units. Hence, the present polymers comprise from 0 to 100%, preferably from 50% to 100%, more preferably from 70% to about 100% of a polymerizable hydrophobic monomer (A, as defined herein after), or mixtures thereof, and from 0 to 100%, preferably from 0% to 50%, more preferably from 0% to about 30% of a polymerizable hydrophilic monomer (B, as defined herein after) or mixtures thereof. Of course, if the polymer comprises both A monomer and B monomer, then the monomers must be copolymerizable with each other.

- The steps and process for making said polymer are those conventionally known to the skilled person and can be as defined above with the exception that the polymer is not grafted to a silicone monomer. Disclosure of these polymers and preparation thereof are disclosed in US 5,120,532.
- The polymer has a weight average molecular weight from about 5,000 to about 1,000,000, preferably from about 30,000 to about 300,000, which has an organic polymeric backbone, said copolymer comprising monomers selected from the group consisting of A monomers, B monomers and mixtures thereof, wherein said copolymer is prepared by the polymerisation combination of the following relative weight percentages of said A monomers, and said B monomers:
 - a- from 0% to about 100%, by weight of said copolymer, of a hydrophobic, A monomer, free radically copolymerizable with said B monomers;
 - b. from 0% to about 100%, by weight of said copolymer, of a hydrophilic reinforcing B monomer, copolymerizable with said A monomer, said B monomer being selected from the group consisting of polar monomers and macromers and mixtures thereof.

The polymers are prepared by the polymerization combination of A monomer, and, if used, B monomer. The copolymer composition is characterized by the amount of each monomer charged to the polymerization reaction vessel. In general, the adhesive polymer will comprise from 0 to 100%, preferably from

WO 00/08249 PCT/US98/16127

about 50% to about 100%, more preferably from about 60% to about 90%, most preferably from about 70% to about 80%, of the hydrophobic, A monomer; and from 0 to 100%, preferably from 0% to about 50.0%, more preferably from 10% to about 40%, most preferably from 20% to 30% of the hydrophilic, reinforcing B monomer.

5

15

20

25

30

Preferably, the polymer of use herein has a molecular weight between about 30,000 and about 150,000.

10 Exemplary polymers for use in the present invention include the following, where the composition of the copolymer is given as weight percentage of each monomer used in the polymerization reaction used to prepare the polymer:

vinyl pyrrolidone/vinyl acetate copolymers (at ratios of up to about 30%, by weight, vinyl pyrrolidone); vinyl acetate homopolymer; t-butyl acrylate homopolymer; t-butyl styrene/ ethyl hexyl methacrylate copolymer (50/50, by weight); dimethyl acrylamide/ t-butyl acrylate/ethyl hexyl methacrylate copolymer (10/45/45); ethylene/vinyl acetate copolymer (12.5/87.5); allyl alcohol/styrene copolymer (19/81); vinyl chloride/vinyl acetate copolymer (83/17 and lower); vinyl pyrrolidone/vinyl acetate/butyl acrylate copolymer (10/78/12 and 10/70/20); vinyl pyrrolidone/ vinyl acetate/butyl acrylate/styrene sulfonate copolymer (10/70/15/5); vinyl pyrrolidone/vinyl propionate copolymer (5/95); vinyl caprolactam/vinyl acetate copolymer (5/95); acrylic acid/t-butyl acrylate (25/75) and styling resins sold under the trade names Ultrahold CA 8.RTM. by Ciba Geigy (ethyl acrylate/ acrylic acid/N-t-butyl acrylamide copolymer), Resyn 28-1310.RTM. by National Starch and Luviset CA 66.RTM. by BASF (vinyl acetate/crotonic acid copolymer 90/10); Luviset CAP.RTM. by BASF (vinyl acetate/vinyl propionate/crotonic acid 50/40/10); and Resyn 28-2930.RTM. by National Starch (vinyl acetate/vinyl neodecanoate/crotonic acid copolymer), and Amerhold DR-25 by Union Carbide (ethyl acrylate/metacrylic acid/methyl methacrylate/acrylic acid copolymer), Poligen A by BASF (polyacrylate dispersion), APPRETAN from Clariant (polyvinyl acetate), MOWEOL from Clariant (polyvinyl alcohol), and SOKALAN EG 310 from BASF (PVP/AA: polyvinylpyrrolidone/acrylic acid).

35 The most preferred polymers among the one disclosed under II are the polymer made of acrylic acid and t-butyl acrylate monomeric units, more preferably with

15

20

25

acrylic acid from about 20% to 30% by weight, and t-butyl acrylate from about 70% to about 80%.

III- Adhesive Block Copolymer

The composition of the present invention comprises select silicone-containing adhesive block copolymers as defined herein, which help provide the improved dewrinkling performance to the composition, and which are soluble or dispersible in the water-solubilising agent of the wrinkle reducing composition.

The silicone-containing adhesive block copolymers selected for use in the wrinkle reducing compositions of the present invention are prepared by the free radical polymerization of select silicone containing macroinitiators (described in detail hereinafter) and ethylenically unsaturated monomers. The resulting block copolymers comprise sequentially arranged moieties or blocks which are further composed of smaller repeating units. The silicone-containing adhesive block copolymers herein comprise silicone-containing blocks derived from silicone-containing macroinitiators (hereinafter Block A) and other blocks derived from ethylenically unsaturated monomers as defined herein (hereinafter Block B). These block copolymers are either A-B block structures containing two block segments; A-B-A block structures containing three block segments, -(A-B)_n-block structures containing multiple blocks wherein n is an integer having a value of 2 or more, or combinations thereof.

The wrinkle reducing compositions of the present invention comprises any one of the of the block structures described above, including mixtures or combinations thereof, and also including combinations thereof with small amounts of unreacted monomer or small amounts of homopolymers derived from the ethylenically unsaturated monomers described herein or the silicone-containing macroinitiators also described herein.

The silicone-containing adhesive block copolymers herein have a weight average molecular weight of from about 10,000 grams/mole to about 10,000,000 grams/mole, preferably from about 20,000 grams/mole to about 1,000,000 grams/mole, more preferably from about 30,000 grams/mole to about 1,000,000 grams/mole, even more preferably from about 60,000 grams/mole to about 750,000 grams/mole, and most preferably from about 70,000 grams/mole and about 750,000 grams/mole.

The silicone-containing adhesive block copolymers for use in the wrinkle reducing compositions herein are prepared by free radical polymerization of ethylenically unsaturated monomers and the silicone-containing macroinitiators described herein. Polymerization reactions of this type are generally well known in the polymer art, some descriptions of which are disclosed by M. Mishra, Macromolecular Design: Concept and Practice, Polymer Frontiers International, Inc., pages 313-358 (1994); European Patent Application 766957A1, published April 9, 1997; and Odian, Principles of Polymerization, 3rd edition, John Wiley & Sons, pages 198-334 (1991), which descriptions are incorporated herein by reference in their entirety.

The free radical polymerization reaction referenced herein can be accomplished, for example, by combining the ethylenically unsaturated monomer and the polysiloxane macroinitiator in a reactor along with a sufficient amount of a mutual solvent so that when the reaction is complete the viscosity of the reaction is reasonable. Undesired terminators, especially oxygen, are removed as needed. This can be done by evacuation or by purging with an inert gas, such as argon or nitrogen. The reaction is brought to the temperature needed for initiation to occur, assuming thermal initiators are used. Alternatively, redox or radiation initiation can be used. The polymerization is allowed to proceed as long as needed for a high level of conversion to be achieved, typically from a few hours to a few days. The solvent is then removed, usually by evaporation or by precipitating the copolymer by addition of a nonsolvent. The resulting block copolymer can be further purified, as desired, and used in formulating the wrinkle reducing composition of the present invention by formulation techniques well known in the art.

The silicone-containing adhesive block copolymers for use herein can also be made by a semi-continuous or continuous process. In the semi-continuous process, two or more additions of monomers or macromonomers are made during the polymerization reaction. This is advantageous when the copolymer is made of several monomers which react during the polymerization at different rates. The proportions of monomers added to the reaction at the separate points of addition can be adjusted by one of ordinary skill in the art such that the polymers of the final product have a more uniform structure. In other words, the

15

25

30

35

polymers of the final product will have a more consistent monomer content distribution for each of the monomer types charged to the reaction.

Other examples of silicone-containing block copolymers and methods of making them are described in U.S. Patent 5,523,365, to Geck et al., issued June 4, 1996; U.S. Patent 4,689,289, to Crivello, issued August 25, 1987; U.S. Patent 4,584,356, to Crivello, issued April 22, 1986; Macromolecular Design, Concept & Practice, Ed: M.K. Mishra, Polymer Frontiers International, Inc., Hopewell Jct., NY (1994); and Block Copolymers, A. Noshay and J.E. McGrath, Academic Press, NY (1977), which descriptions are incorporated herein by reference.

Silicone-containing Macroinitiator

The silicone-containing adhesive block copolymers of the wrinkle reducing composition herein comprise from about 2% to about 50%, preferably from about 5% to about 40%, and more preferably from about 10% to about 30%, by weight of silicone-containing blocks, wherein the silicone-containing blocks are derived from the select silicone-containing macroinitiators described herein.

The silicone-containing macroinitiator for use in the wrinkle reducing composition of the present invention is selected from the group consisting of the following formulas:

or combinations thereof, wherein each R is independently selected from the group consisting of C1-C10 alkyl, phenyl, C1-C10 alkyl-substituted phenyl, and mixtures thereof, preferably methyl group; each X is a divalent linking group which may be the same or different for any given silicone-containing macroazoinitiator, and which may include the following groups: acyl, alkyl, aryl, amide, alkene, alkyne, ether, ester, sulfone, sulfoxide, thioether, halogen, nitrile and combinations thereof, preferably amide or ester groups for ease of synthesis. Other divalent linking groups can also be used provided that they to not substantially and unduly impair the free radical polymerization reactions described herein.

In the above described macroinitiator formulas, each Y is independently selected from the group consisting of those chemical moieties represented by the following formulas:

10

30

or combinations thereof; each of m, n, and p value are positive integers each independently having a value of 1 or greater, preferably m and p have values independently from about 14 to about 700, whereas n has no upper value except that it should not be so large as to limit applicability for practical reasons (viscosity, processing, solvent compatibility, etc.) during the polymerization reaction of the silicone-containing adhesive block copolymers, but is preferably a value of from about 1 to about 10.

- The number average molecular weight of silicone-containing macroinitiator is from about 500 grams/mole to about 500,000 grams/mole, preferably from about 2,000 grams/mole to about 250,000 grams/mole, more preferably from about 5,000 grams/mole to about 100,000 grams/mole.
- 20 Preferred are the silicone-containing macroinitiators wherein Y is an azo group, even more preferably those silicone-containing macroazoinitiators corresponding to the following formula:

wherein x is an integer having a value of from about 50 to about 150, and n is an integer having a value of from about 4 to about 12, more preferably from about 6 to about 9. Most preferred are those macroazoinitiators represented by the above formula wherein x is an integer having a value of either about 135 or 67, and n is an integer having a value of from about 6 to about 9.

The silicone-containing blocks (Block A) of the block copolymers herein preferably contain at least about 10 repeating monomer units, more preferably at least about 40 monomer units, even more preferably at least about 60 monomer

10

15

units, wherein each of the repeating monomer units are derived from the select silicone-containing macroinitiators described herein, and the average molecular weight of each silicone-containing block preferably ranges from about 500 grams/mole to about 60,000 grams/mole, more preferably from about 1,000 grams/mole to about 25,000 grams/mole, even more preferably from about 2,000 grams/mole to about 15,000 grams/mole.

Ethylenically Unsaturated Monomer

The silicone-containing adhesive block copolymers of the wrinkle reducing composition herein comprise from about 50% to about 98%, preferably from about 60% to about 95%, and more preferably from about 70% to about 90%, by weight of a copolymerizable ethylenically unsaturated monomers.

The blocks (block B) in the silicone-containing adhesive block copolymer are derived from ethylenically unsaturated monomers, wherein block B has a glass transition temperature (Tg value) of more than about -20°C, more preferably more than about -5°C, and also preferably less than about 60°C, more preferably less than about 50°C, and even more preferably less than about 40°C.

The ethylenically unsaturated monomers are copolymerizable with the silicone-containing macroinitiators and contain at least one polymerizable carbon-carbon double bond, which can be mono-, di-, tri- or tetra-substituted. Preferred are vinyl monomers. Either a single type of ethylenically unsaturated monomer or combination of two or more ethylenically unsaturated monomers can be used.
The ethylenically unsaturated monomers are selected to meet the requirements of or preferences for the silicone-containing adhesive block copolymers described herein.

The ethylenically unsaturated monomers for use in making the siliconecontaining adhesive block copolymers may be hydrophilic or hydrophobic, water soluble or water insoluble. These ethylenically unsaturated monomers are preferably hydrophilic monomers, or combinations of hydrophilic and hydrophobic monomers provided that the resulting block copolymer in the wrinkle reducing composition has the requisite solubility and other characteristics defined herein. The ethylenically unsaturated monomers when copolymerized into repeating units or blocks (block B) in the silicone-containing adhesive block copolymer herein preferably contains at least about 10 repeating monomers, more preferably at least about 20 repeating monomers, even more preferably at least about 50 repeating monomer units.

Nonlimiting classes of ethylenically unsaturated monomers useful herein include unsaturated alcohols. unsaturated monocarboxylic acids. unsaturated dicarboxylic acids, unsaturated anhydrides, alcohol esters of unsaturated monocarboxylic acids, alcohol esters of unsaturated dicarboxylic acids, alcohol unsaturated anhydrides, alkoxylated esters of unsaturated esters of monocarboxylic acids, alkoxylated esters of unsaturated dicarboxylic acids, alkoxylated esters of unsaturated anhydrides, aminoalkyl esters of unsaturated monocarboxylic acids, aminoalkyl esters of unsaturated dicarboxylic acids, esters of unsaturated anhydrides, amides of unsaturated aminoalkvl monocarboxylic acids, amides of unsaturated dicarboxylic acids, amides of unsaturated anhydrides, salts of unsaturated monocarboxylic acids, salts of unsaturated dicarboxylic acids, salts of unsaturated anhydrides, unsaturated hydrocarbons, unsaturated heterocycles, and combinations thereof.

20

25

30

35

5

10

15

Some examples of suitable ethylenically unsaturated monomers include, but are not limited to, acrylic acid, methacrylic acid, N,N-dimethylacrylamide, dimethylaminoethyl methacrylate, quaternized dimethylaminoethyl methacrylate, methacrylamide, N-t-butyl acrylamide, maleic acid, maleic anhydride and its half esters, crotonic acid, itaconic acid, acrylamide, acrylate alcohols, hydroxyethyl methacrylate, diallyldimethyl ammonium chloride, vinyl pyrrolidone, vinyl ethers (such as methyl vinyl ether), maleimides, vinyl pyridine, vinyl imidazole, other polar vinyl heterocyclics, styrene sulfonate, allyl alcohol, vinyl alcohol (such as that produced by the hydrolysis of vinyl acetate after polymerization), vinyl caprolactam, acrylic and methacrylic acid esters of C₁-C₁₈ alcohols, such as methanol, ethanol, methoxy ethanol, 1-propanol, 2-propanol, 1-butanol, 2-methyl-1-propanol, 1-pentanol, 2-pentanol, 3-pentanol, 2-methyl-1-butanol, 1-methyl-1butanol, 3-methyl-1-butanol, 1-methyl-1-pentanol, 2-methyl-1-pentanol, 3-methyl-1-pentanol, t-butanol(2-methyl-2-propanol), cyclohexanol, neodecanol, 2-ethyl-1butanol, 3-heptanol, benzyl alcohol, 2-octanol, 6-methyl-1-heptanol, 2-ethyl-1hexanol, 3,5-dimethyl-1-hexanol, 3,5,5-tri methyl-1-hexanol, 1-decanol, 1WO 00/08249 PCT/US98/16127

dodecanol, 1-hexadecanol, 1-octa decanol, and the like, the alcohols preferably having from about 1 to about 12 carbon atoms; dicyclopentenyl acrylate; 4-biphenyl acrylate; pentachlorophenyl acrylate; 3,5-dimethyladamantyl acrylate; 3,5-dimethyladamentyl methacrylate; 4-methoxycarbonylphenyl methacrylate; trimethylsilyl methacrylate; styrene; alkyl substituted styrenes including alphamethylstyrene and t-butylstyrene; vinyl esters, including vinyl acetate, vinyl neononanoate, vinyl pivalate and vinyl propionate; vinyl chloride; vinylidene chloride; vinyl toluene; alkyl vinyl ethers, including isobutyl vinyl ether and \underline{s} -butyl vinyl ether; butadiene; cyclohexadiene; bicycloheptadiene; 2,3-dicarboxylmethyl-1,6-hexadiene; ethylene; propylene; indene; norbornylene; β -pinene; α -pinene; salts of acids and amines listed above, and combinations thereof. The quaternized monomers can be quaternized either before or after the free radical copolymerization reaction with the macroinitiator described hereinafter.

15 Preferred ethylenically unsaturated monomers include acrylic acid, methacrylic acid, N,N-dimethyl acrylamide, dimethylaminoethyl methacrylate, quaternized dimethylaminoethyl methacrylate, vinyl pyrrolidone, acrylic or methacrylic acid esters of C₁-C₁₈ alcohols, styrene, alpha-methylstyrene, t-butylstyrene, vinyl acetate, vinyl propionate, 2-methoxyethyl acrylate, 2-ethoxyethyl acrylate, isobutyl methacrylate, t-butyl acrylate, t-butyl methacrylate, 2-ethylhexyl methacrylate, methyl methacrylate, salts of any acids and amines listed above, and combinations thereof.

Neutralized Block Copolymers

5

10

The silicone-containing adhesive block copolymers may comprise acidic functionalities, such as carboxyl groups, and are usually used in at least partially neutralized form to promote solubility or dispensability of the block copolymer in water. In addition, use of the neutralized form aids in the ability of the compositions to be removed from the fabrics by washing. The extent of such neutralization ranges from about 10% to 100%, more preferably from about 20% to about 90%, even more preferably from about 40% to about 85%, neutralization of the acidic functionalities of the silicone-containing adhesive block copolymer.

35 Neutralization of the silicone-containing adhesive block copolymer containing acidic functionalities may be accomplished by any conventional or otherwise

10

15

20

25

30

known technique for affecting such neutralization by using an organic or inorganic base material. Metallic bases are particularly useful for this purpose. Suitable base neutralizers include, but are not limited to, ammonium hydroxides, alkali metal hydroxides, or an alkaline earth metal hydroxides, preferably potassium hydroxide and sodium hydroxide. Examples of other suitable neutralizing agents include, but are not limited to, amines or amino alcohols such as 2-amino-2-methyl-1,3-propanediol (AMPD), 2-amino-2-ethyl-1,3-propanediol (AEPD), 2-amino-2-methyl-1-propanol (AMP), 2-amino-1-butanol (AB), monoethanolamine (MEA), diethanolamine (DEA), triethanolamine (TEA), monoisopropanolamine (MIPA), diisopropanolamine (DIPA), triisopropanolamine (TIPA) and dimethyl stearamine (DMS) and combinations thereof. Preferred are amines and metallic bases.

Neutralization of silicone-containing adhesive block copolymer containing basic functionalities, e.g., amino groups, are likewise preferably at least partially neutralized with an organic or inorganic acid e.g., hydrogen chloride. Neutralization can be accomplished by any conventional or otherwise known technique for accomplishing such neutralization. The preferred extent of neutralization is the same as that described for neutralization of acidic functionalities.

Solubility of the selected silicone-containing adhesive block copolymer, if the selected copolymer contains an acidic or basic functionality, should be determined after the desired acid or base neutralization.

Preferred Silicone-Containing Adhesive Block Copolymers

Nonlimiting examples of preferred silicone-containing adhesive block polymers include, but are not limited to, the following block copolymers derived from the macroazoinitiators which conform to the following formula.

[Poly(dimethyl siloxane)-block-poly(t-butyl acrylate-co-n-butyl acrylate-co-acrylic acid-co-methacrylic acid)]n

30

t-butyl acrylate: 40%; n-butyl acrylate: 28%; acrylic acid: 12%; methacrylic acid: 10%;

silicone macroazoinitiator (above-described formula, x=135, n is from about 6 to about 9) 10%;

5 Molecular weight of silicone block: 10,000 grams/mole Polymer molecular weight:114,000 grams/mole.

[Poly(dimethyl siloxane)-block-poly(t-butyl acrylate-co-acrylic acid)]n t-butyl acrylate: 40%; n-butyl acrylate: 24%; acrylic acid: 21%;

silicone macroazoinitiator (above-described formula, x=135, n is from about 6 to about 9)15%;

Molecular weight of silicone block: 10,000 grams/mole.

Polymer molecular weight:86,000 grams/mole.

15 [Poly(dimethyl siloxane)-block-poly(t-butyl acrylate-co-ethyl acrylate-co-acrylic acid)]n

t-butyl acrylate: 32%; ethyl acrylate: 33%; acrylic acid: 20%;

silicone macroazoinitiator (above-described formula, x=67, n is from about 6-9) 15%.

20 Polymer molecular weight: 110,600 grams/mole.

Molecular weight of silicone block: 5000 grams/mole.

The preferred silicone-containing macroazoinitiators described above are available from Wako Chemical USA, Inc., Richmond, Virginia, USA as VPS1001 and VPS 0501 (poly(dimethylsiloxane)initiators).

Also useful herein are sulfur-linked silicone containing copolymers, including block copolymers. As used herein in reference to silicone containing copolymers, the term "sulfur-linked" means that the copolymer contains a sulfur linkage (i.e., -S-), a disulfide linkage (i.e., -S-S-), or a sulfhydryl group (i.e., -SH).

These sulfur-linked silicone-containing copolymers are represented by the following general formula:

$$G_{1}$$
 G_{2}
 G_{2}
 G_{2}
 G_{3}
 G_{4}
 G_{6}
 G_{3}
 G_{3}
 G_{3}
 G_{3}
 G_{3}
 G_{4}
 G_{4}
 G_{4}
 G_{4}
 G_{4}

wherein

5

10

15

20

25

30

each G_5 and G_6 is independently selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, fluoroalkyl, hydrogen, and —ZSA, wherein A represents a vinyl polymeric segment consisting essentially of polymerized free radically polymerizable monomer, and Z is a divalent linking group (Useful divalent linking groups Z include but are not limited to the following: C_1 to C_{10} alkylene, alkarylene, arylene, and alkoxyalkylene. Preferably, Z is selected from the group consisting of methylene and propylene for reasons of commercial availability.);

each G2 comprises A;

each G₄ comprises A;

each R₁ is a monovalent moiety selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, fluoroalkyl, hydrogen, and hydroxyl (Preferably, R₁ represents monovalent moieties which can independently be the same or different selected from the group consisting of C₁₋₄ alkyl and hydroxyl for reasons of commercial availability. Most preferably, R₁ is methyl.);

each R_2 is a divalent linking group (Suitable divalent linking groups include but are not limited to the following: C_1 to C_{10} alkylene, arylene, alkarylene, and alkoxyalkylene. Preferably, R_2 is selected from the group consisting of C_{1-3} alkylene and C_7 - C_{10} alkarylene due to ease of synthesis of the compound. Most preferably, R_2 is selected from the group consisting of — CH_2 —, 1,3-propylene, and

$$-CH_2-CH_2CH_2-$$

each R₃ represents monovalent moieties which can independently be the same or different and are selected from the group consisting of alkyl, aryl, alkaryl, alkoxy, alkylamino, fluoroalkyl, hydrogen, and hydroxyl (Preferably, R₃ represents monovalent moieties which can independently be the same or different selected from the group consisting of C₁₋₄ alkyl and hydroxyl for reasons of commercial availability. Most preferably, R₃ is methyl.);

each R_4 is a divalent linking group(Suitable divalent linking groups include but are not limited to the following: C_1 to C_{10} alkylene, arylene, alkarylene, and alkoxyalkylene. Preferably, R_4 is selected from the group consisting of C_{1-3} alkylene and C_7 - C_{10} alkarylene for ease of synthesis. Most preferably, R_4 is selected from the group consisting of — CH_2 —, 1,3-propylene, and

WO 00/08249 PCT/US98/16127

31

$$-CH_2-CH_2CH_2-$$

x is an integer of 0-3;

y is an integer of 5 or greater(preferably y is an integer ranging from about 14 to about 700, preferably from about 20 to about 200); and

g is an integer of 0-3;

wherein at least one of the following is true:

q is an integer of at least 1;

x is an integer of at least 1;

G₅ comprises at least one —ZSA moiety; or

10 G₆ comprises at least one —ZSA moiety.

As noted above, A is a vinyl polymeric segment formed from polymerized free radically polymerizable monomers. The selection of A is typically based upon the intended uses of the composition, and the properties the copolymer must possess in order to accomplish its intended purpose. If A comprises a block in the case of block copolymers, a polymer having AB and/or ABA architecture will be obtained depending upon whether a mercapto functional group —SH is attached to one or both terminal silicon atoms of the mercapto functional silicone compounds, respectively. The weight ratio of vinyl polymer block or segment, to silicone segment of the copolymer can vary. The preferred copolymers are those wherein the weight ratio of vinyl polymer segment to silicone segment ranges from about 98:2 to 50:50, in order that the copolymer possesses properties inherent to each of the different polymeric segments while retaining the overall polymer's solubility.

25

15

20

5

Sulfur linked silicone copolymers are described in more detail in U.S. Patent No. 5,468,477, to Kumar et al., issued November 21, 1995, and PCT Application No. WO 95/03776, assigned to 3M, published February 9, 1995, which are incorporated by reference herein in their entirety.

30

Of course, the term "adhesive polymer" can also encompass mixed adhesive polymers.

The adhesive polymer is present in the composition in a sufficient amount to result in an amount of from 0.01% to 1%, preferably from 0.01% to 0.5%, more preferably from 0.01% to 0.1% by weight of polymer per weight of dry fabrics.

- Typically, the adhesive polymer is present in the composition of the present 5 invention in an amount of from 0.05% to about 5.0%, preferably from about 0.1% to about 2.0%, more preferably from about 0.2% to about 1.0% by weight of the composition.
- It is not intended to exclude the use of higher or lower levels of the polymers, as 10 long as an effective amount is used to provide adhesive and film-forming properties to the composition and the composition can be formulated and effectively applied for its intended purpose.

15 B-Water-solubilising agent

30

35

A water-solubilising agent is another essential ingredient of the invention. Indeed, it provides the sufficient solubilisation of the polymer but also further ensures an effective plasticizing of the polymer resulting in a flexible polymer network.

- 20 Any type of water-soluble surfactant can be used to impart the solubilising property. However, some water-soluble surfactants and mixtures thereof are more preferred. Hence, it is preferred that the surfactant is a surface-active molecule with a linear or branched hydrophobic tail and a positively charged hydrophilic head group. More preferably, the water-soluble solubilising agents for 25 use herein are selected from water-soluble anionic surfactant, water-soluble nonionic surfactant, and mixtures thereof.
 - By "water-soluble solubilising agent", it is meant that the solubilising agent forms either a dispersion or substantially clear, isotropic solutions when dissolved in water at 0.2 weight percent at 25°C.

Water-soluble nonionic surfactant

Still suitable solubilising agents are the nonionic surfactants. Typical of these surfactants are the alkoxylated surfactants, which provide a low surface tension that permits the composition to spread readily and more uniformly on hydrophobic surfaces like polyester and nylon. Said surfactant is preferably

10

15

20

25

30

included when the composition is used in a spray dispenser in order to enhance the spray characteristics of the composition and allow the composition to distribute more evenly, and to prevent clogging of the spray apparatus. The spreading of the composition also allows it to dry faster, so that the treated material is ready to use sooner. For concentrated compositions, the surfactant facilitates the dispersion of many actives such as antimicrobial actives and perfumes in the concentrated aqueous compositions.

Nonlimiting examples of nonionic alkoxylated surfactants include addition products of ethylene oxide with fatty alcohols, fatty acids, fatty amines, etc. Optionally, addition products of propylene oxide with fatty alcohols, fatty acids, fatty amines may be used.

Suitable compounds are surfactants of the general formula:

$$R^2 - Y - (C_2H_4O)_z - C_2H_4OH$$

wherein R² is selected from the group consisting of primary, secondary and branched chain alkyl and/or acyl hydrocarbyl groups; primary, secondary and branched chain alkenyl hydrocarbyl groups; and primary, secondary and branched chain alkyl- and alkenyl-substituted phenolic hydrocarbyl groups; said hydrocarbyl groups preferably having a hydrocarbyl chain length of from 6 to 20, preferably from 8 to 18 carbon atoms. More preferably the hydrocarbyl chain length is from 10 to 18 carbon atoms. In the general formula for the ethoxylated nonionic surfactants herein, Y is -O-, -C(O)O-, -C(O)N(R)-, or -C(O)N(R)R-, in which R, when present, is R² or hydrogen, and z is at least 2, preferably at least 4, more preferably from 5 to 11.

The nonionic surfactants herein are characterised by an HLB (hydrophilic-lipophilic balance) of from 7 to 20, preferably from 8 to 15. Of course, by defining R² and the number of ethoxylate groups, the HLB of the surfactant is, in general, determined. However, it is to be noted that the nonionic ethoxylated surfactants useful herein contain relatively long chain R² groups and are relatively highly ethoxylated. While shorter alkyl chain surfactants having short ethoxylated groups may possess the requisite HLB, they are not as effective herein.

10

15

20

25

Examples of nonionic surfactants follow. The nonionic surfactants of this invention are not limited to these examples. In the examples, the integer defines the number of ethoxyl (EO) groups in the molecule.

a. Straight-Chain, Primary Alcohol Alkoxylates

The tri-, penta-, hepta-ethoxylates of dodecanol, and tetradecanol are useful surfactants in the context of this invention. The ethoxylates of mixed natural or synthetic alcohols in the "coco" chain length range are also useful herein. Commercially available straight-chain, primary alcohol alkoxylates for use herein are available under the tradename Marlipal® 24/70, Marlipal 24/100, Marlipal 24/150 from Huls, and Genapol® C-050 from Hoechst.

b. Straight-Chain, Secondary Alcohol Alkoxylates

The tri-, penta-, hepta-ethoxylates of 3-hexadecanol, 2-octadecanol, 4-eicosanol, and 5-eicosanol are useful surfactants in the context of this invention.

A commercially available straight-chain secondary alcohol ethoxylate for use herein is the material marketed under the tradename Tergitol 15-S-7 from Union Carbide, which comprises a mixture of secondary alcohols having an average hydrocarbyl chain length of 11 to 15 carbon atoms condensed with an average 7 moles of ethylene oxide per mole equivalent of alcohol. Still another suitable commercially available straight-chain secondary alcohol ethoxylate for use herein is the material marketed under the tradename Softanol obtainable from BP Chemicals Ltd. or Nippon Catalytic of Japan. Particularly useful herein are Softanol 50, Softanol 90, which comprises a mixture of linear secondary alcohol having an average hydrocarbyl chain length of 11 to 16 carbon atoms condensed with an average of 5 to 10 moles of ethylene oxide per mole equivalent of alcohol.

c. Alkyl Phenol Alkoxylates

30 Suitable alkyl phenol alkoxylates are the polyethylene oxide condensates of alkyl phenols, e.g., the condensation products of alkyl phenols having an alkyl or alkenyl group containing from 6 to 20 carbon atoms in a primary, secondary or branched chain configuration, preferably from 8 to 12 carbon atoms, with ethylene oxide, the said ethylene oxide being preferably present in amounts appear of alkyl phenol. The alkyl phenol of alkyl phenol. The alkyl

WO 00/08249 PCT/US98/16127

substituent in such compounds may be derived from polymerized propylene, diisobutylene, octane, and nonane.

35

Examples of this type of nonionic surfactants include Triton N-57® a nonyl phenol ethoxylate (5EO) from Rohm & Haas, Dowfax® 9N5 from Dow and Lutensol® AP6 from BASF.

5

10

20

d. Olefinic Alkoxylates

The alkenyl alcohols, both primary and secondary, and alkenyl phenols corresponding to those disclosed immediately hereinabove can be ethoxylated and used as surfactants.

Commercially available olefinic alkoxylates for use herein are available under the tradename Genapol O-050 from Hoechst.

e. Branched Chain Alkoxylates

Branched chain primary and secondary alcohols (or Guerbet alcohols) which may be available from the well-known "OXO" process or modification thereof can be ethoxylated.

Particularly preferred among these ethoxylates of the primary OXO alcohols are the surfactants marketed under the name Lutensol by BASF or Dobanol by the Shell Chemicals, U.K., LTD. The preferred Dobanols are primary alcohols with hydrocarbyl groups of 9 to 15 carbon atoms, with the majority having a hydrocarbyl group of 13 carbon atoms. Particularly preferred are Dobanols with an average degree of ethoxylation of 3 to 11, and preferably 7 on the average.

An example of this type of material is an aliphatic alcohol ethylene oxide condensate having from 3 to less than 9 moles of ethylene oxide per mole of aliphatic alcohol, the aliphatic alcohol fraction having from 9 to 14 carbon atoms. Other examples of this type of nonionic surfactants include certain of the commercially available Dobanol®, Neodol® marketed by Shell, Lutensol® from BASF, or Lial® from Enichem. For example Dobanol® 23.5 (C12-C13 EO5), Dobanol® 91.5 (C9-C11 EO 5), Neodol 45 E5, and Lial-145.7 EO (oxo C 14 15 alcohol + 7.0 mol of EO), Lial 111 EO 6 and Isalchem 123 series from Enichem.

Other suitable nonionic alkoxylated surfactants are alkyl amines alkoxylated with at least 5 alkoxy moieties. Typical of this class of compounds are the surfactants derived from the condensation of ethylene oxide with an hydrophobic alkyl amine

product. Preferably the hydrophobic alkyl group, has from 6 to 22 carbon atoms. Preferably, the alkyl amine is alkoxylated with 10 to 40, and more preferably 20 to 30 alkoxy moieties.

- 5 Example of this type of nonionic surfactants are the alkyl amine ethoxylate commercially available under the tradename Genamin from Hoechst. Suitable example for use herein are Genamin C-100, Genamin O-150, and Genamin S-200.
- 10 Still other suitable type of nonionic surfactant among this class are the N,N',N'-polyoxyethylene (12)-N-tallow 1,3 diaminopropane commercialised under the tradename Ethoduomeen T22 from Akzo, and Synprolam from ICI.

Further suitable nonionic surfactants are the alkyl amide surfactants.

15

20

25

30

35

Further nonlimiting examples of nonionic alkoxylated surfactants include the surfactant which are cyclodextrin-compatible, that is it should not substantially form a complex with the cyclodextrin so as to diminish performance of the cyclodextrin and/or the surfactant. Complex formation diminishes both the ability of the cyclodextrin to absorb odors and the ability of the surfactant to lower the surface tension of the aqueous composition. This include block copolymers of ethylene oxide and propylene oxide. Suitable block polyoxyethylenepolyoxypropylene polymeric surfactants, are suitable ingredients when a cyclodextrin is present. Indeed, it will further increase the compatibility of the polymer with the cyclodextrin. Typical of these surfactants that are compatible with most cyclodextrins, include those based on ethylene glycol, propylene glycol, glycerol, trimethylolpropane and ethylenediamine as the initial reactive Polymeric compounds made from a sequential hydrogen compound. ethoxylation and propoxylation of initial compounds with a single reactive hydrogen atom, such as C₁₂₋₁₈ aliphatic alcohols, are not generally compatible with the cyclodextrin. Certain of the block polymer surfactant compounds designated Pluronic® and Tetronic® by the BASF-Wyandotte Corp., Wyandotte, Michigan, are readily available.

Non limiting examples of surfactants of this type include:

WO 00/08249 PCT/US98/16127

37

<u>Pluronic Surfactants</u> with the general formula $H(EO)_n(PO)_m(EO)_nH$,

wherein EO is an ethylene oxide group, PO is a propylene oxide group, and n and m are numbers that indicate the average number of the groups in the surfactants. Typical examples of cyclodextrin-compatible Pluronic surfactants

5 are:

10

Name	Average MW	Average n	Average m
L-44	2,200	10	23
L-43	1,850	6	22
F-38	4,700	43	16
P-84	4,200	19	43,

and mixtures thereof.

<u>Tetronic Surfactants</u> with the general formula:

$$H(EO)n(PO)m (PO)m(EO)nH \\ H(EO)n(PO)m ((PO)m(EO)nH$$

wherein EO, PO, n, and m have the same meanings as above. Typical examples of cyclodextrin-compatible Tetronic surfactants are:

	Name	Average MW	Average n	Average m	
20	901	4,700	3	18	
	908	25,000	114	22 ,	
	and mixtures thereof.				

25 "Reverse" Pluronic and Tetronic surfactants have the following general formulas:

Reverse Pluronic Surfactants H(PO)_m(EO)_n(PO)_mH

30 Reverse Tetronic Surfactants

wherein EO, PO, n, and m have the same meanings as above. Typical examples of Reverse Pluronic and Reverse Tetronic surfactants are:

Reverse Pluronic surfactants:

5	Name	Average MW	Average n	Average m
	10 R5	1,950	8	22
	25 R1	2,700	21	6
	Reverse Te	tronic surfactants		
	Name	Average MW	Average n	Average m
10	130 R2	7,740	9	26
	70 R2	3,870	4	13

The silicone surfactants

A preferred class of nonionic alkoxylated surfactants are the polyalkyleneoxide polysiloxanes having a dimethyl polysiloxane hydrophobic moiety and one or more hydrophilic polyalkylene side chains. Examples of this type of surfactants are the Silwet[®] surfactants which are available OSi Specialties, Inc., Danbury, Connecticut, and have the general formula:

$$(CH_3)_3SiO$$
 $(SiO)a$
 $(SiO)b$
 $(CH_3)_3$
 CH_3
 CH_3
 CH_3
 CH_3

20

wherein a + b are from about 1 to about 50, preferably from about 3 to about 30, more preferably from about 10 to about 25, and R^1 is mainly one or more random poly(ethyleneoxide/propyleneoxide) copolymer groups having the general formula:

wherein n is 3 or 4, preferably 3; total c (for all polyalkyleneoxy side groups) has a value of from 1 to about 100, preferably from about 6 to about 100; total d is from 0 to about 14, preferably from 0 to about 3; and more preferably d is 0; total c+d has a value of from about 5 to about 150, preferably from about 9 to about 100 and each R² is the same or different and is selected from the group consisting of hydrogen, an alkyl having 1 to 4 carbon atoms, and an acetyl group, preferably hydrogen and methyl group.

Representative Silwet surfactants are as follows.

	Name	Average MW	Average a+b	Average total c
	L-7608	600	1	9
	L-7607	1,000	2	17
	L-77	600	1	9
5	L-7605	6,000	20	99
	L-7604	4,000	21	53
	L-7600	4,000	11	68
	L-7657	5,000	20	76
	L-7602	3,000	20	29

The molecular weight of the polyalkyleneoxy group (R¹) is less than or equal to about 10,000. Preferably, the molecular weight of the polyalkyleneoxy group is less than or equal to about 8,000, and most preferably ranges from about 300 to about 5,000. Thus, the values of c and d can be those numbers which provide molecular weights within these ranges. However, the number of ethyleneoxy units (-C₂H₄O) in the polyether chain (R¹) must be sufficient to render the polyalkyleneoxide polysiloxane water dispersible or water soluble. If propyleneoxy groups are present in the polyalkylenoxy chain, they can be distributed randomly in the chain or exist as blocks. Preferred Silwet surfactants are L-7600, L-7602, L-7604, L-7605, L-7657, and mixtures thereof. Besides surface activity, polyalkyleneoxide polysiloxane surfactants can also provide other benefits, such as antistatic benefits, lubricity and softness to fabrics.

The preparation of polyalkyleneoxide polysiloxanes is well known in the art. Polyalkyleneoxide polysiloxanes of the present invention can be prepared according to the procedure set forth in U.S. Pat. No. 3,299,112, incorporated herein by reference. Typically, polyalkyleneoxide polysiloxanes of the surfactant blend of the present invention are readily prepared by an addition reaction between a hydrosiloxane (i.e., a siloxane containing silicon-bonded hydrogen) and an alkenyl ether (e.g., a vinyl, allyl, or methallyl ether) of an alkoxy or hydroxy end-blocked polyalkylene oxide). The reaction conditions employed in addition reactions of this type are well known in the art and in general involve heating the reactants (e.g., at a temperature of from about 85° C. to 110° C.) in the presence of a platinum catalyst (e.g., chloroplatinic acid) and a solvent (e.g., toluene).

The above ethoxylated nonionic surfactants are useful in the present process invention alone or in combination, and the term "nonionic surfactant" encompasses mixed nonionic surface active agents.

5 Still further nonionic surfactants which may be of use herein are the polyhydroxyfatty acid surfactants as described in EP-A-659870.

Preferred nonionic surfactants for use herein are the addition products of ethylene oxide with fatty alcohols. Among this class of surfactants, commercially preferred surfactants are those available under the tradenames Marlipal 24/100, Marlipal 24/150, Lial 111 EO 6, and Softanol 90. Branched surfactants are most preferred.

Water-soluble anionic surfactant

15 Still suitable solubilising agents are the anionic surfactants. Suitable anionic surfactants for the purpose of the invention include the alkyl sulphates (RSO₄), alkyl ether sulphates (R(OCH2CH2)eSO4), alkyl sulphonates (RSO3) such as sodium paraffin sulphonate, alkyl succinates (ROOCCH2CH2COOZ), alkyl carboxylates (RCOOM), alkyl ether carboxylates (R(OCH₂CH₂)_eCOOM). In the formulae in brackets, R is a hydrophobic chain (C6-C22) alkyl or alkenyl, e is 20 from 0 to 20, Z is M or R', M is H or any counterion such as those known in the art, including Na, K, Li, NH₄, amine, and R' is a C₁-C₅ alkyl group, possibly functionalized with hydroxyl groups, preferably C₁-C₃, most preferably methyl. Still another suitable anionic surfactant is coco imino dipropionate. Still other 25 preferred anionic surfactants for use herein are the alkyl sulphosuccinates (R'OOCCH₂CH(SO₃M)COOR') wherein R' is a hydrophobic chain (C₆-C₁₈, preferably C₈-C₁₂) linear or branched alkyl or alkenyl, and M is as defined hereinbefore. Preferred alkyl sulphosuccinates are commercially available from CYTEC Industries under the tradename Aerosol OT, and Aerosol AOT. Preferred 30 among the above described anionic surfactants are selected from the alkyl sulphate surfactants, alkyl sulphonate surfactants, alkyl sulphosuccinate surfactants, and mixtures thereof. Preferred alkyl sulphates for use herein are selected from sodium dodecyl alkyl sulphate, sodium tallow alkyl sulphate, sodium lauryl sulphate, sodium octyl sulphate and mixtures thereof. Preferred 35 commercially available compounds are Empicol® 0298/F and/or Empimin® LV33 from Albright and Wilson.

Still another preferred anionic surfactant for use herein has the general formula:

wherein R is an alkyl group. Examples of this type of surfactants are available from the Dow Chemical Company under the trade name Dowfax $^{(\!R\!)}$ wherein R is a linear or branched C_6 - C_{16} alkyl group. An example of these cyclodextrin-compatible anionic surfactant is Dowfax 3B2 with R being approximately a linear C_{10} group. These anionic surfactants are preferably not used when the antimicrobial active or preservative, etc., is cationic to minimize the interaction with the cationic actives, since the effect of both surfactant and active are diminished.

Preferred surfactants for use herein are the surfactant which will form clear composition comprising the polymers. Preferred surfactants for this purpose are the nonionic surfactants which are herein above described as addition products of ethylene oxide with fatty alcohols. Among this class of surfactants, commercially preferred surfactants are those available under the tradenames Marlipal 24/100, Marlipal 24/150, Lial 111 EO 6, and Softanol 90. More preferred are the branched nonionic surfactants of the above class.

20

5

10

15

The solubilising agent is present in the composition in a sufficient amount to solubilise the polymer. Accordingly, the solubilising agent is present in an amount of from 20 to 500% by weight, preferably from 50% to 200% by weight of the polymer concentration. Most preferably, it has been found that the solubilisation of the polymer is most effective when the concentration of the solubilising agent is substantially equal to that of the polymer. Accordingly, the concentration of the solubilising agent is most preferably from 75% to 150% by weight of the polymer concentration.

30

25

However, for the purpose of the invention, the total level of solubilising agent in the composition is in an amount of less than 5% by weight, preferably from 0.1 to less than 5%, more preferably from 0.1% to 2.0% by weight of the

composition. By use of this surfactant, a sufficient wetting and solubilisation of the polymer is obtained. Furthermore, when a cyclodextrin is present, the above amount does not hinder the efficacity of the cyclodextrin.

5 C. <u>Liquid Carrier</u>

The liquid carrier used in the composition of the present invention is an aqueous system comprising water. Optionally, but not preferably, in addition to the water, the carrier can contain a low molecular weight organic solvent that is highly soluble in water, e.g., C₁-C₄ monohydric alcohols, alkylene carbonates, and mixtures thereof. Examples of these water-soluble solvents include ethanol, propanol, isopropanol, etc. Water is the main liquid carrier due to its low cost, availability, safety, and environmental compatibility. Water can be distilled, deionized, or tap water.

The level of liquid carrier in the compositions of the present invention is typically greater than 80%, preferably greater than 90%, more preferably greater than 95%, by weight of the composition. When a concentrated composition is used, the level of liquid carrier is typically from 50% to 95%, by weight of the composition, preferably from 60% to 97%, more preferably from 70% to 99%, by weight of the composition.

D. Optional Ingredients

1- Nonionic polyhydric alcohol humectant

A nonionic humectant of the polyhydric alcohol type is a preferred optional ingredient of the composition of the present invention. Typical of these compounds are the low molecular weight polyols.

Low molecular weight polyols with relatively high boiling points, as compared to water, are essential ingredients of the composition of the present invention.

By "low molecular weight", it is meant that the compounds preferably have a molecular weight below 1000, preferably from 50 to 500, more preferably from 55 to 200.

Preferably, these polyols are short chain. By "short chain ", it is meant that the compounds have a carbon chain length of less than 10 carbon atoms, preferably less than 8 carbon atoms.

Not to be bound by theory, it is believed that the incorporation of a small amount of nonionic polyhydric alcohol humectant into the compositions containing the solubilising agent reinforces the hydrogen breaking process as well as reducing the fabric drying rate, thereby allowing more time to the fabric to relax, and also deliver a basic level of lubrication and plasticizing of the polymer.

10

Preferred polyols for use herein are selected from polyols having from 2 to 8 hydroxy groups.

Preferably the glycol used is glycerol, ethylene glycol, propylene glycol, diethylene glycol, dipropylene glycol, sorbitol, erythritol or mixtures thereof, more preferably diethylene glycol, ethylene glycol, propylene glycol, dipropylene glycol and mixtures thereof.

Some polyols, e.g., dipropylene glycol, are also useful to facilitate the solubilization of some perfume ingredients in the composition of the present invention. Both diethylene glycol and dipropylene glycol are favored for use herein as it provides non-stickiness properties on hard surfaces and/or fabrics.

The humectant is present in the composition in a sufficient amount to result in an amount of from 0.005% to 5%, preferably from 0.01% to 3%, more preferably from 0.01% to 1.50% by weight of active per weight of dry fabrics.

Typically, the humectant is added to the composition of the present invention at a level of from about 0.01% to about 10%, by weight of the composition, preferably from about 0.1% to about 3%, more preferably from about 0.1% to about 1.5%, by weight of the composition.

2-Lubricant

30

35

In addition to the above humectants which can impart a lubrication property to the fabrics, the composition may also optionally employ typical lubricating compounds. Lubricants have also been found beneficial in providing "wrinkles resistance" of dry-cleaned fabrics.

Typical lubricants are those conventionally known as softeners and include the cationic softener and nonionic softeners.

Cationic softeners

Typical of the cationic softening components are the quaternary ammonium compounds or amine precursors thereof as defined hereinafter.

10

A)-Quaternary Ammonium Fabric Softening Active Compound

(1) Preferred quaternary ammonium fabric softening active compound have the formula

$$\left[(R) \frac{+}{4-m} N \left[(CH_2)_n - Q - R^1 \right]_m \right] X^{-1}$$
(1)

15

or the formula:

$$\begin{bmatrix} (R)_{4m} & + & \\ (CH_2)_n & - & CH & - & CH_2 & - & Q & - & R^1 \end{bmatrix}_m X$$

$$Q = R^1 \qquad (2)$$

wherein Q is a functional unit having the formula:

20

25

each R unit is independently hydrogen, C_1 - C_6 alkyl, C_1 - C_6 hydroxyalkyl, and mixtures thereof, preferably methyl or hydroxy alkyl; each R¹ unit is independently linear or branched C_{11} - C_{22} alkyl, linear or branched C_{11} - C_{22} alkenyl, and mixtures thereof, R² is hydrogen, C_1 - C_4 alkyl, C_1 - C_4 hydroxyalkyl, and mixtures thereof; X is an anion which is compatible with fabric softener actives and adjunct ingredients; the index m is from 1 to 4, preferably 2.

An example of a preferred fabric softener active is a mixture of quaternized amines having the formula:

$$R_2 - N = (CH_2)_n - O - C - R^1$$
 X^-

wherein R is preferably methyl; R¹ is a linear or branched alkyl or alkenyl chain comprising at least 11 atoms, preferably at least 15 atoms. In the above fabric softener example, the unit -R¹ represents a fatty alkyl or alkenyl unit which is typically derived from a triglyceride source. The triglyceride source is preferably derived from tallow, partially hydrogenated tallow, lard, partially hydrogenated lard, vegetable oils and/or partially hydrogenated vegetable oils, such as, canola oil, safflower oil, peanut oil, sunflower oil, corn oil, soybean oil, tall oil, rice bran oil, etc. and mixtures of these oils.

The preferred fabric softening actives of the present invention are the Diester and/or Diamide Quaternary Ammonium (DEQA) compounds, the diesters and diamides having the formula:

$$\begin{bmatrix} (R)_{\overline{4-m}} + \begin{bmatrix} (CH_2)_n - Q - R^{1} \end{bmatrix}_m \end{bmatrix} X^{-1}$$

wherein R, R¹, X, and n are the same as defined herein above for formulas (1) and (2), and Q has the formula:

These preferred fabric softening actives are formed from the reaction of an amine with a fatty acyl unit to form an amine intermediate having the formula:

$$R = N \left[(CH_2)_n - Q - R^1 \right]_2$$

25

wherein R is preferably methyl, Q and R¹ are as defined herein before; followed by quaternization to the final softener active.

Non-limiting examples of preferred amines which are used to form the DEQA fabric softening actives according to the present invention include methyl bis(2-hydroxyethyl)amine having the formula:

5

$$HO$$
 N
 OH

methyl bis(2-hydroxypropyl)amine having the formula:

$$_{\text{HO}}$$
 $\stackrel{\text{CH}_3}{\stackrel{\text{I}}{\longrightarrow}}$
 $_{\text{OH}}$

10 methyl (3-aminopropyl) (2-hydroxyethyl)amine having the formula:

methyl bis(2-aminoethyl)amine having the formula:

$$H_{2N}$$
 N
 N
 N
 N

15

triethanol amine having the formula:

$$\text{HO} \overset{\text{N}}{\searrow} \text{OH}$$

di(2-aminoethyl) ethanolamine having the formula:

20

$$H_2N$$
 OH NH_2

The counterion, $X^{(-)}$ above, can be any softener-compatible anion, preferably the anion of a strong acid, for example, chloride, bromide, methylsulfate,

ethylsulfate, sulfate, nitrate and the like, more preferably chloride or methyl sulfate. The anion can also, but less preferably, carry a double charge in which case $X^{(-)}$ represents half a group.

Tallow and canola oil are convenient and inexpensive sources of fatty acyl units which are suitable for use in the present invention as R¹ units. The following are non-limiting examples of quaternary ammonium compounds suitable for use in the compositions of the present invention. The term "tallowyl" as used herein below indicates the R¹ unit is derived from a tallow triglyceride source and is a mixture of fatty alkyl or alkenyl units. Likewise, the use of the term canolyl refers to a mixture of fatty alkyl or alkenyl units derived from canola oil.

In the following table are described non-limiting examples of suitable fabric softener according to the above formula. In this list, the term "oxy" defines a

15 — unit, whereas the term "oxo" defines a -O- unit.

Table II

Fabric Softener Actives

N,N-di(tallowyl-oxy-2-oxo-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium

20 chloride;

35

N,N-di(canolyl-oxy-2-oxo-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium chloride;

N,N-di(tallowyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride;

N,N-di(canolyloxy-2-oxo-ethyl)-N,N-dimethyl ammonium chloride

25 N,N,N-tri(tallowyl-oxy-2-oxo-ethyl)-N-methyl ammonium chloride;

N,N,N-tri(canolyl-oxy-2-oxo-ethyl)-N-methyl ammonium chloride;

N-(tallowyloxy-2-oxo-ethyl)-N-(tallowyl)-N,N-dimethyl ammonium chloride;

N-(canolyloxy-2-oxo-ethyl)-N-(canolyl)-N,N-dimethyl ammonium chloride;

1,2-di(tallowyloxy-oxo)-3-N,N,N-trimethylammoniopropane chloride; and

30 1,2-di(canolyloxy-oxo)-3-N,N,N-trimethylammoniopropane chloride; and mixtures of the above actives.

Other examples of quaternay ammoniun softening compounds are methylbis(tallowamidoethyl)(2-hydroxyethyl)ammonium methylsulfate and methylbis(hydrogenated tallowamidoethyl)(2-hydroxyethyl)ammonium

methylsulfate; these materials are available from Witco Chemical Company under the trade names Varisoft® 222 and Varisoft® 110, respectively.

Particularly preferred is N,N-di(tallowyl-oxy-2-oxo-ethyl)-N-methyl, N-(2-hydroxyethyl) ammonium chloride, where the tallow chains are at least partially unsaturated.

The level of unsaturation contained within the tallow, canola, or other fatty acyl unit chain can be measured by the lodine Value (IV) of the corresponding fatty acid, which in the present case should preferably be in the range of from 5 to 100 with two categories of compounds being distinguished, having a IV below or above 25.

Indeed, for compounds having the formula:

15

20

25

10

5

$$\begin{bmatrix} (R)_{\overline{4-m}} & + \\ (CH_2)_m - Q - R^1 \end{bmatrix}_m X^{-1}$$

derived from tallow fatty acids, when the lodine Value is from 5 to 25, preferably 15 to 20, it has been found that a *cis/trans* isomer weight ratio greater than about 30/70, preferably greater than about 50/50 and more preferably greater than about 70/30 provides optimal concentrability.

For compounds of this type made from tallow fatty acids having a lodine Value of above 25, the ratio of *cis* to *trans* isomers has been found to be less critical unless very high concentrations are needed.

Other suitable examples of fabric softener actives are derived from fatty acyl groups wherein the terms "tallowyl" and canolyl" in the above examples are replaced by the terms "cocoyl, palmyl, lauryl, oleyl, ricinoleyl, stearyl, palmityl," which correspond to the triglyceride source from which the fatty acyl units are derived. These alternative fatty acyl sources can comprise either fully saturated, or preferably at least partly unsaturated chains.

30

As described herein before, R units are preferably methyl, however, suitable fabric softener actives are described by replacing the term "methyl" in the above examples in Table II with the units "ethyl, ethoxy, propyl, propoxy, isopropyl, butyl, isobutyl and t-butyl.

í

15

The counter ion, X, in the examples of Table II can be suitably replaced by bromide, methylsulfate, formate, sulfate, nitrate, and mixtures thereof. In fact, the anion, X, is merely present as a counterion of the positively charged quaternary ammonium compounds. The scope of this invention is not considered limited to any particular anion.

Mixtures of actives of formula (1) and (2) may also be prepared.

2)-Still other suitable quaternary ammonium fabric softening compounds for use herein are cationic nitrogenous salts having two or more long chain acyclic aliphatic C₈-C₂₂ hydrocarbon groups or one said group and an arylalkyl group which can be used either alone or as part of a mixture are selected from the group consisting of:

(i) acyclic quaternary ammonium salts having the formula:

$$\begin{bmatrix} R^4 \\ R^8 - N - R^5 \\ R^8 \end{bmatrix}^+ A^-$$

- wherein R⁴ is an acyclic aliphatic C₈-C₂₂ hydrocarbon group, R⁵ is a C₁-C₄ saturated alkyl or hydroxyalkyl group, R⁸ is selected from the group consisting of R⁴ and R⁵ groups, and A- is an anion defined as above;
 - (ii) diamino alkoxylated quaternary ammonium salts having the formula:

$$\begin{bmatrix} O & R^{5} & O \\ R^{1}-C-NH-R^{2}-N-R^{2}-NH-C-R^{1} \\ (CH_{2}CH_{2}O)_{n}H \end{bmatrix}^{+} A^{-}$$

wherein n is equal to 1 to about 5, and R^1 , R^2 , R^5 and A^- are as defined above; (iii) mixtures thereof.

25

10

25

Examples of the above class cationic nitrogenous salts are the well-known dialkyldi methylammonium salts such as ditallowdimethylammonium chloride, ditallowdimethylammonium methylsulfate,

di(hydrogenatedtallow)dimethylammonium chloride, distearyldimethylammonium chloride, dibehenyldimethylammonium chloride. Di(hydrogenatedtallow)di methylammonium chloride and ditallowdimethylammonium chloride are preferred. Examples of commercially available dialkyldimethyl ammonium salts usable in the present invention are di(hydrogenatedtallow)dimethylammonium chloride (trade name Adogen® 442), ditallowdimethylammonium chloride (trade name Adogen® 3445), distearyl dimethylammonium chloride (trade name Arosurf® TA-100), all available from Witco Chemical Company. Dibehenyldimethylammonium chloride is sold under the trade name Kemamine Q-2802C by Humko Chemical Division of Witco Chemical Corporation.

Dimethylstearylbenzyl ammonium chloride is sold under the trade names Varisoft

8 SDC by Witco Chemical Company and Ammonyx® 490 by Onyx Chemical
Company.

B)-Amine Fabric Softening Active Compound

Suitable amine fabric softening compounds for use herein, which may be in amine form or cationic form are selected from:

(i)- Reaction products of higher fatty acids with a polyamine selected from the group consisting of hydroxyalkylalkylenediamines and dialkylenetriamines and mixtures thereof. These reaction products are mixtures of several compounds in view of the multi-functional structure of the polyamines.

The preferred Component (i) is a nitrogenous compound selected from the group consisting of the reaction product mixtures or some selected components of the mixtures.

One preferred component (i) is a compound selected from the group consisting of substituted imidazoline compounds having the formula:

10

15

20

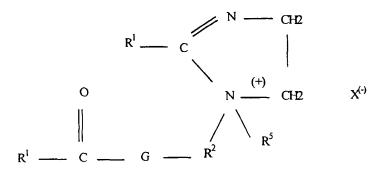
25

wherein R^7 is an acyclic aliphatic $C_{15}\text{-}C_{21}$ hydrocarbon group and R^8 is a divalent $C_1\text{-}C_3$ alkylene group.

Component (i) materials are commercially available as: Mazamide® 6, sold by Mazer Chemicals, or Ceranine® HC, sold by Sandoz Colors & Chemicals; stearic hydroxyethyl imidazoline sold under the trade names of Alkazine® ST by Alkaril Chemicals, Inc., or Schercozoline® S by Scher Chemicals, Inc.; N,N"-ditallowalkoyldiethylenetriamine; 1-tallowamidoethyl-2-tallowimidazoline (wherein in the preceding structure R^1 is an aliphatic C_{15} - C_{17} hydrocarbon group and R^8 is a divalent ethylene group).

N,N"-ditallowalkoyldiethylenetriamine 1-tallow(amidoethyl)-2-Both and tallowimidazoline аге reaction products of tallow fatty acids diethylenetriamine, and are precursors of the cationic fabric softening agent methyl-1-tallowamidoethyl-2-tallowimidazolinium methylsulfate (see "Cationic Surface Active Agents as Fabric Softeners," R. R. Egan, Journal of the American Oil Chemicals' Society, January 1978, pages 118-121). N,N"-ditallow alkoyldiethylenetriamine and 1-tallowamidoethyl-2-tallowimidazoline can be obtained from Witco Chemical Company as experimental chemicals. Methyl-1tallowamidoethyl-2-tallowimidazolinium methylsulfate is sold by Witco Chemical Company under the tradename Varisoft® 475.

(ii)-softener having the formula:



wherein each R^2 is a C_{1-6} alkylene group, preferably an ethylene group; and G is an oxygen atom or an -NR- group; and each R, R^1 , R^2 and R^5 have the definitions given above and A⁻ has the definitions given above for X⁻.

10

15

20

An example of Compound (ii) is 1-oleylamidoethyl-2-oleylimidazolinium chloride wherein R^1 is an acyclic aliphatic C_{15} - C_{17} hydrocarbon group, R^2 is an ethylene group, G is a NH group, R^5 is a methyl group and A^- is a chloride anion.

(iii)- softener having the formula:

wherein R, R¹, R², and A⁻ are defined as above.

An example of Compound (iii) is the compound having the formula:

wherein R¹ is derived from oleic acid.

Further suitable cationic softeners for use herein are the cationic silicones such as described in GB-1,549,180, and EP-A-0,450,815.

Nonionic softeners

Nonionic softener include compounds such as the fatty acid esters, preferably a partial ester, of mono- or polyhydric alcohols or anhydride thereof containing from 1 to 8 carbon atoms.

It is preferred that the fatty acid ester has at least 1 free (i.e. unesterified) hydroxyl group and at least 1 fatty acyl group.

The mono- or polyhydric alcohol portion of the ester can be represented by methanol, isobutanol, 2-ethyl hexanol, isopropanol, ethylene glycol and polyethylene glycol with a maximum of 5 ethylene glycol units, glycerol, diglycerol, xylitol, sucrose, erythritol, penta-erythritol, sorbitol or sorbitan. Ethylene glycol, glycerol and sorbitan esters are particularly preferred.

The fatty acid portion of the ester normally comprises a fatty acid having from 12 to 22 carbon atoms, typical examples being lauric acid, myristic acid, palmitic acid, stearic acid and behenic acid.

5

10

One highly preferred group of lubricant for use in the present invention is the sorbitan esters, which are esterified dihydration products of sorbitol. Sorbitol, itself prepared by the catalytic hydrogenation of glucose, can be dehydrated in well known fashion to form mixtures of 1,4- and 1,5-sorbitol anhydrides and small amounts of isosorbides. (See Brown, U.S. Pat. No. 2,322,821, issued June 29, 1943). The foregoing type of complex mixtures of anhydrides of sorbitol are collectively referred to herein as "sorbitan". It will be recognized that this "sorbitan" mixture will also contain some free, uncyclized sorbitol.

15

The lubricants of the type employed herein can be prepared by esterifying the "sorbitan" mixture with a fatty acyl group in standard fashion, e.g. by reaction with a fatty acid halide or fatty acid. The esterification reaction can occur at any of the available hydroxyl groups, and various mono-, di-, etc., esters can be prepared. In fact, mixtures of mono-, di-, tri-, etc., esters almost always result from such reactions, and the stoichiometric ratios of the reactants can be simply adjusted to favor the desired reaction product.

25

20

For commercial production of the sorbitan ester materials, etherification and esterification are generally accomplished in the same processing step by reacting sorbitol directly with fatty acids. Such a method of sorbitan ester preparation is described more fully in MacDonald; "Emulsifiers: Processing and Quality Control:", Journal of the American Oil Chemists' Society, Volume 45, October 1968.

30

The mixtures of hydroxy-substituted sorbitan esters useful herein contain, inter alia, compounds of the following formulae, as well as the corresponding hydroxy-substituted di-esters:

10

15

wherein the group R is a C10-C26, and higher, fatty alkyl residue. Preferably this fatty alkyl residue contains from 16 to 22 carbon atoms. The fatty alkyl residue can, of course, contain non-interfering substituents such as hydroxyl groups. Esterified hydroxyl groups can, of course, be either in terminal or internal positions within the sorbitan molecule.

The foregoing complex mixtures of esterified dehydration products of sorbitol (and small amounts of esterified sorbitol) are collectively referred to herein as "sorbitan esters". Sorbitan mono- and di-esters of lauric, myristic, palmitic, stearic and behenic (docosanoic) acids are particularly useful herein as softening agents and also can provide an anti-static benefit to fabrics. Mixed sorbitan esters, e.g. mixtures of the foregoing esters, and mixtures prepared by esterifying sorbitan with fatty acid mixtures such as the mixed tallow fatty acids, are useful herein and are economically attractive. Unsaturated C10-C22 sorbitan esters, e.g. sorbitan monooleate, usually are present in such mixtures in low concentration.

20

35

The term "alkyl" as employed herein to describe the sorbitan esters encompasses both the saturated and unsaturated hydrocarbyl ester side chain groups.

5 Certain derivatives of the sorbitan esters herein, especially the "lower" ethoxylates thereof (i.e. mono-, di- and tri-esters) wherein one or more of the unesterified --OH groups contain one to about 20 oxyethylene moieties (Tweens®) are also useful in the composition of the present invention. Therefore, for purposes of the present invention, the term "sorbitan ester" includes such derivatives.

Preparation of the sorbitan esters can be achieved by dehydrating sorbitol to form a mixture of anhydrides of the type set forth above, and subsequently esterifying the mixture using, for example, a 1:1 stoichiometry for the esterification reaction. The esterified mixture can then be separated into the various ester components. Separation of the individual ester products is, however, difficult and expensive.

Accordingly, it is easier and more economical not to separate the various esters, using instead the esterified mixture as the sorbitan ester component. Such mixtures of esterified reaction products are commercially available under various tradenames e.g. Span® Such sorbitan ester mixtures can also be prepared by utilizing conventional interesterification procedures.

For the purposes of the present invention, it is preferred that a significant amount of di- and tri-sorbitan esters are present in the ester mixture. Ester mixtures having from 20%-50% mono-ester, 25% to 50% di-ester and 10%-35% of tri- and tetra-esters are preferred. The material which is sold commercially as sorbitan mono-ester (e.g. mono-stearate) does in fact contain significant amounts of di- and tri-esters and a typical analysis of sorbitan monostearate indicates that it comprises ca.27% mono-, 32% di- and 30% tri- and tetra esters. Commercial sorbitan mono-stearate therefore is a preferred material.

Mixtures of sorbitan stearate and sorbitan palmitate having stearate/palmitate weight ratios varying between 10:1 and 1:10, and 1,5-sorbitan esters are useful. Both the 1,4- and 1,5- sorbitan esters are useful herein. Other useful alkyl

20

35

sorbitan esters for use in the softening compositions herein include sorbitan monolaurate, sorbitan monomyristate, sorbitan monopolmitate, sorbitan monopolmitate, sorbitan monopolate, sorbitan dilaurate, sorbitan dipalmitate, sorbitan

distearate, sorbitan dibehenate, sorbitan dioleate, and mixtures thereof, and mixed tallowalkyl sorbitan mono- and di-esters. Such mixtures are readily prepared by reacting the foregoing hydroxy-substituted sorbitans, particularly the 1,4- and 1,5-sorbitans, with the corresponding acid or acid chloride in a simple esterification reaction, It is to be recognized, of course, that commercial materials prepared in this manner will comprise mixtures usually containing minor proportions of uncyclized

sorbitol, fatty acids, polymers, isosorbide structures, and the like.

It is also to be recognized that the sorbitan esters employed herein can contain up to about 15% by weight of esters of the C20 -C26, and higher, fatty acids, as well as minor amounts of C8, and lower, fatty esters.

Other fatty acid partial esters useful in the present invention are xylitol monopalmitate, pentaerythritol monostearate, sucrose monostearate, glycerol monostearate and ethylene glycol monostearate. As with the sorbitan esters, commercially available mono-esters normally contain substantial quantities of dior tri-esters.

The glycol esters are also highly preferred. These are the mono-, di- or tri-esters of glycerol and fatty acids of the class described above.

Commercial glyceryl monostearate, which may contain a proportion of the di- and tristearates, is especially preferred.

30 Another class of suitable nonionic lubricants are the cyclomethicones such as described in EP636356.

The above-discussed nonionic compounds are correctly termed "lubricating agents", because, when the compounds are correctly applied to a fabric, they do impart a soft, lubricious feel to the fabric.

Additional fabric softening agents useful herein are described in U.S. Pat. No. 4,661,269, issued April 28, 1987, in the names of Toan Trinh, Errol H. Wahl, Donald M. Swartley, and Ronald L. Hemingway; U.S. Pat. No. 4,439,335, Burns, issued March 27, 1984; and in U.S. Pat. Nos.: 3,861,870, Edwards and Diehl; 4,308,151, Cambre; 3,886,075, Bernardino; 4,233,164, Davis; 4,401,578, Verbruggen; 3,974,076, Wiersema and Rieke; 4,237,016, Rudkin, Clint, and Young; and European Patent Application publication No. 472,178, by Yamamura et al., all of said documents being incorporated herein by reference.

10 Of course, the term " lubricating agent " can also encompass mixed softening active agents.

Preferred lubricating agent among the one disclosed above are the quaternary ammonium compound disclosed under (A) and the cyclomethicones.

15

The lubricating agent is present in the composition in a sufficient amount to result in an amount of from 0.005% to 5%, preferably from 0.01% to 3%, more preferably from 0.01% to 1.50% by weight of active per weight of dry fabrics.

Typically, the lubricating agent is added to the composition of the present invention at a level of from about 0.01% to about 10%, by weight of the composition, preferably from about 0.1% to about 3%, more preferably from about 0.1% to about 1.5%, by weight of the composition.

25 **3- Salt**

A salt is another optional ingredient. When used, it will both contribute to the hydrogen bond breaking process produces by the water and to the reinforcement of the wetting power of the wetting agent. The salt is further believed to facilitate the dewrinkling action by maintaining a residual humidity of fibers.

30

35

The salt useful in the present invention is a compound made of alkaline and/or earth alkaline metal, and that can form hydrates upon crystallization. Typically, the salt for use in the present invention have the following formula: AM; wherein A is a cation. This cation is an alkaline and/or earth alkaline metal,

preferably selected from sodium, calcium, potassium, magnesium; more preferably sodium and calcium, and

10

20

25

30

wherein M is a couteranion selected from sulfate, chloride, nitrate, carbonate, borate, and carboxylates.

Preferred salts are salts selected from sodium, calcium, potassium, magnesium and mixtures thereof; more preferably salt of sodium, calcium, and mixtures thereof.

Particularly preferred salts for use herein are selected from sodium sulphate, sodium bicarbonate, sodium chloride, sodium borate, potassium sulphate, calcium chloride, sodium citrate, magnesium sulphate, and mixtures thereof, more preferably are selected from sodium sulphate, sodium bicarbonate, potassium sulphate, calcium chloride, and mixtures thereof.

The salt is present in the composition in a sufficient amount to result in an amount of from 0.005% to 5%, preferably from 0.01% to 3%, more preferably from 0.01% to 1.50% by weight of active per weight of dry fabrics.

Accordingly, typical levels of the salt in the composition are from 0.01% to about 10%, by weight of the composition, preferably from about 0.1% to about 3%, more preferably from about 0.1% to about 1.5%, by weight of the composition.

4- Cyclodextrin

In a preferred aspect of the invention, the composition of the invention comprises an optional cyclodextrin. This will impart the composition with odour absorbing properties, which is especially useful for application on inanimate surfaces to control the malodour, whilst not being detrimental to the dewrinkling performance of the composition.

Furthermore, when a cyclodextrin is employed in the composition of the invention, it has been found that the silicone grafted adhesive polymer is not compatible therewith. It has now been found that the nonionic water-solubilising agent, preferably the branched chain surfactants described herein before described, which is employed in the compositions of the invention provides a beneficial effect on the cyclodextrin compatibility with the polymer.

10

15

20

25

30

35

As used herein, the term "cyclodextrin" includes any of the known cyclodextrins such as unsubstituted cyclodextrins containing from six to twelve glucose units. especially, alpha-cyclodextrin, beta-cyclodextrin, gamma-cyclodextrin and/or their derivatives and/or mixtures thereof. The alpha-cyclodextrin consists of six glucose units, the beta-cyclodextrin consists of seven glucose units, and the gamma-cyclodextrin consists of eight glucose units arranged in donut-shaped rings. The specific coupling and conformation of the glucose units give the cyclodextrins a rigid, conical molecular structures with hollow interiors of specific volumes. The "lining" of each internal cavity is formed by hydrogen atoms and glycosidic bridging oxygen atoms; therefore, this surface is fairly hydrophobic. The unique shape and physical-chemical properties of the cavity enable the cyclodextrin molecules to absorb (form inclusion complexes with) organic molecules or parts of organic molecules which can fit into the cavity. Many odorous molecules can fit into the cavity including many malodorous molecules and perfume molecules. Therefore, cyclodextrins, and especially mixtures of cyclodextrins with different size cavities, can be used to control odors caused by a broad spectrum of organic odoriferous materials, which may, or may not, contain reactive functional groups. The complexation between cyclodextrin and odorous molecules occurs rapidly in the presence of water. However, the extent of the complex formation also depends on the polarity of the absorbed molecules. In an aqueous solution, strongly hydrophilic molecules (those which are highly water-soluble) are only partially absorbed, if at all. Therefore, cyclodextrin does not complex effectively with some very low molecular weight organic amines and acids when they are present at low levels on wet fabrics. As the water is being removed however, e.g., the fabric is being dried off, some low molecular weight organic amines and acids have more affinity and will complex with the cyclodextrins more readily.

The cavities within the cyclodextrin in the solution of the present invention should remain essentially unfilled (the cyclodextrin remains uncomplexed) while in solution, in order to allow the cyclodextrin to absorb various odor molecules when the solution is applied to a surface. Non-derivatised (normal) beta-cyclodextrin can be present at a level up to its solubility limit of about 1.85% (about 1.85g in 100 grams of water) at room temperature. Beta-cyclodextrin is not preferred in compositions which call for a level of cyclodextrin higher than its water solubility limit. Non-derivatised beta-cyclodextrin is generally not preferred when the

10

15

20

25

30

35

composition contains surfactant since it affects the surface activity of most of the preferred surfactants that are compatible with the derivatized cyclodextrins.

Preferably, the solution of the present invention is clear. The term "clear" as defined herein means transparent or translucent, preferably transparent, as in "water clear," when observed through a layer having a thickness of less than about 10 cm.

Preferably, the cyclodextrins for use herein are highly water-soluble such as, alpha-cyclodextrin and/or derivatives thereof, gamma-cyclodextrin and/or derivatives thereof, derivatised beta-cyclodextrins, and/or mixtures thereof. The derivatives of cyclodextrin consist mainly of molecules wherein some of the OH groups are converted to OR groups. Cyclodextrin derivatives include, e.g., those with short chain alkyl groups such as methylated cyclodextrins, and ethylated cyclodextrins, wherein R is a methyl or an ethyl group; those with hydroxyalkyl substituted groups, such as hydroxypropyl cyclodextrins and/or hydroxyethyl cyclodextrins, wherein R is a -CH₂-CH(OH)-CH₃ or a -CH₂CH₂-OH group; branched cyclodextrins such as maltose-bonded cyclodextrins; cationic cyclodextrins such as those containing 2-hydroxy-3-(dimethylamino)propyl ether, wherein R is CH₂-CH(OH)-CH₂-N(CH₃)₂ which is cationic at low pH; quaternary ammonium, e.g., 2-hydroxy-3-(trimethylammonio)propyl ether chloride groups, wherein R is CH₂-CH(OH)-CH₂-N⁺(CH₃)₃Cl⁻; anionic cyclodextrins such as carboxymethyl cyclodextrins, cyclodextrin sulfates, and cyclodextrin succinylates; amphoteric cyclodextrins such as carboxymethyl/quaternary ammonium cyclodextrins; cyclodextrins wherein at least one glucopyranose unit has a 3-6anhydro-cyclomalto structure, e.g., the mono-3-6-anhydrocyclodextrins, as disclosed in "Optimal Performances with Minimal Chemical Modification of Cyclodextrins", F. Diedaini-Pilard and B. Perly, The 7th International Cyclodextrin Symposium Abstracts, April 1994, p. 49, said references being incorporated herein by reference; and mixtures thereof. Other cyclodextrin derivatives are disclosed in U.S. Pat. Nos: 3,426,011, Parmerter et al., issued Feb. 4, 1969; 3,453,257; 3,453,258; 3,453,259; and 3,453,260, all in the names of Parmerter et al., and all issued July 1, 1969; 3,459,731, Gramera et al., issued Aug. 5, 1969; 3,553,191, Parmerter et al., issued Jan. 5, 1971; 3,565,887, Parmerter et al., issued Feb. 23, 1971; 4,535,152, Szejtli et al., issued Aug. 13, 1985; 4,616,008, Hirai et al., issued Oct. 7, 1986; 4,678,598, Ogino et al., issued Jul. 7, 1987;

10

15

20

25

4,638,058, Brandt et al., issued Jan. 20, 1987; and 4,746,734, Tsuchiyama et al., issued May 24, 1988; all of said patents being incorporated herein by reference.

Highly water-soluble cyclodextrins are those having water solubility of at least about 10 g in 100 ml of water at room temperature, preferably at least about 20 g in 100 ml of water, more preferably at least about 25 g in 100 ml of water at room temperature. The availability of solubilized, uncomplexed cyclodextrins is essential for effective and efficient odor control performance. Solubilized, water-soluble cyclodextrin can exhibit more efficient odor control performance than non-water-soluble cyclodextrin when deposited onto surfaces, especially fabric.

Examples of preferred water-soluble cyclodextrin derivatives suitable for use herein are hydroxypropyl alpha-cyclodextrin, methylated alpha-cyclodextrin, methylated beta-cyclodextrin, hydroxyethyl beta-cyclodextrin, and hydroxypropyl beta-cyclodextrin. Hydroxyalkyl cyclodextrin derivatives preferably have a degree of substitution of from about 1 to about 14, more preferably from about 1.5 to about 7, wherein the total number of OR groups per cyclodextrin is defined as the degree of substitution. Methylated cyclodextrin derivatives typically have a degree of substitution of from about 1 to about 18, preferably from about 3 to about 16. A known methylated beta-cyclodextrin is heptakis-2,6-di-O-methyl-βcyclodextrin, commonly known as DIMEB, in which each glucose unit has about 2 methyl groups with a degree of substitution of about 14. A preferred, more commercially available, methylated beta-cyclodextrin is a randomly methylated beta-cyclodextrin, commonly known as RAMEB, having different degrees of substitution, normally of about 12.6. RAMEB is more preferred than DIMEB, since DIMEB affects the surface activity of the preferred surfactants more than RAMEB. The preferred cyclodextrins are available, e.g., from Cerestar USA, Inc. and Wacker Chemicals (USA), Inc.

It is also preferable to use a mixture of cyclodextrins. Such mixtures absorb odors more broadly by complexing with a wider range of odoriferous molecules having a wider range of molecular sizes. Preferably at least a portion of the cyclodextrins is alpha-cyclodextrin and its derivatives thereof, gamma-cyclodextrin and its derivatives thereof, and/or derivatised beta-cyclodextrin, more preferably a mixture of alpha-cyclodextrin, or an alpha-cyclodextrin derivative, and derivatised beta-cyclodextrin, even more preferably a mixture of

derivatised alpha-cyclodextrin and derivatised beta-cyclodextrin, most preferably a mixture of hydroxypropyl alpha-cyclodextrin and hydroxypropyl beta-cyclodextrin, and/or a mixture of methylated alpha-cyclodextrin and methylated beta-cyclodextrin.

5

10

It is further believed that a small amount of low molecular weight polyol as defined herein before into the composition comprising the uncomplexed cyclodextrin enhances the formation of the cyclodextrin inclusion complexes as the fabric dries. Further, the incorporation of such polyol provides an improved odor control performance of the composition of the present invention comprising said cyclodextrin.

It is believed that the polyols' ability to remain on the fabric for a longer period of time than water, as the fabric dries allows it to form ternary complexes with the cyclodextrin and some malodorous molecules. The addition of the glycols is believed to fill up void space in the cyclodextrin cavity that is unable to be filled by some malodor molecules of relatively smaller sizes.

20

15

Cyclodextrin compositions prepared by processes that result in a level of such polyols are highly desirable, since they can be used without removal of the polyols.

25

Diethylene glycol is particularly useful in the presence of the uncomplexd cyclodextrin. Indeed, it has been found to enhance the removal of small malodour molecules.

The preferred weight ratio of low molecular weight cyclodextrin to polyol is from about 50:1 to about 1:11, more preferably from about 20:1 to about 1:1, even more preferably from about 10:1 to about 1:1, and most preferably from about 5:1 to about 1:1.

35

30

For controlling odor on fabrics, the composition is preferably used as a spray. It is preferable that the usage compositions of the present invention contain low levels of cyclodextrin so that a visible stain does not appear on the fabric at normal usage levels. Preferably, the solution used to treat the surface under usage conditions is virtually not discernible when dry. Typical levels of

10

15

20

cyclodextrin in usage compositions for usage conditions are from about 0.01% to about 5%, preferably from about 0.1% to about 4%, more preferably from about 0.2% to about 2% by weight of the composition. Compositions with higher concentrations can leave unacceptable visible stains on fabrics as the solution evaporates off of the fabric. This is especially a problem on thin, colored, synthetic fabrics. In order to avoid or minimize the occurrence of fabric staining, it is preferable that the fabric be treated at a level of less than about 5 mg of cyclodextrin per gram of fabric, more preferably less than about 2 mg of cyclodextrin per gram of fabric. The presence of the surfactant can improve appearance by minimizing localized spotting.

Concentrated compositions can also be used in order to deliver a less expensive product. When a concentrated product is used, i.e., when the level of cyclodextrin used is from about 3% to about 20%, more preferably from about 5% to about 10%, by weight of the concentrated composition, it is preferable to dilute the concentrated composition before treating fabrics in order to avoid staining. Preferably the concentrated cyclodextrin composition is diluted with about 50% to about 6000%, more preferably with about 75% to about 2000%, most preferably with about 100% to about 1000% by weight of the concentrated composition of water. The resulting diluted compositions have usage concentrations of cyclodextrin as discussed hereinbefore, e.g., of from about 0.1% to about 5%, by weight of the diluted composition.

5- Antimicrobial active

- The composition may suitably use an optional solubilized, water-soluble antimicrobial active, useful in providing protection against organisms that become attached to the treated material. The free, uncomplexed antimicrobial, e.g., antibacterial, active provides an optimum antibacterial performance.
- 30 Sanitization of fabrics can be achieved by the compositions of the present invention containing, antimicrobial materials, e.g., antibacterial halogenated compounds, quaternary compounds, and phenolic compounds.
- Biguanides. Some of the more robust antimicrobial halogenated compounds which can function as disinfectants/sanitizers as well as finish product preservatives (vide infra), and are useful in the compositions of the present

invention include 1,1'-hexamethylene bis(5-(p-chlorophenyl)biguanide), commonly known as chlorhexidine, and its salts, e.g., with hydrochloric, acetic and gluconic acids. The digluconate salt is highly water-soluble, about 70% in water, and the diacetate salt has a solubility of about 1.8% in water. When chlorhexidine is used as a sanitizer in the present invention it is typically present at a level of from about 0.001% to about 0.4%, preferably from about 0.002% to about 0.3%, and more preferably from about 0.01% to about 0.1%, by weight of the usage composition. In some cases, a level of from about 1% to about 2% may be needed for virucidal activity.

10

15

Other useful biguanide compounds include Cosmoci[®] CQ[®], Vantocil[®] IB, including poly (hexamethylene biguanide) hydrochloride. Other useful cationic antimicrobial agents include the bis-biguanide alkanes. Usable water soluble salts of the above are chlorides, bromides, sulfates, alkyl sulfonates such as methyl sulfonate and ethyl sulfonate, phenylsulfonates such as p-methylphenyl sulfonates, nitrates, acetates, gluconates, and the like.

Examples of suitable bis biguanide compounds are chlorhexidine; 1,6-bis-(2ethylhexylbiguanidohexane)dihydrochloride; 1,6-di-(N₁,N₁'-phenyldiguanido-20 N₅,N₅')-hexane tetrahydrochloride; 1,6-di-(N₁,N₁'-phenyl-N₁,N₁'methyldiguanido-N5,N5')-hexane dihydrochloride; 1,6-di(N₁,N₁'-ochlorophenyldiguanido-N5,N5')-hexane dihydrochloride; 1,6-di(N₁,N₁'-2,6dichlorophenyldiguanido-N5,N5')hexane dihydrochloride; 1,6-di[N1,N1'-.beta.-(pdiguanido-N₅,N₅']-hexane dihydrochloride; 1,6-di(N₁,N₁'methoxyphenyl) 25 .alpha.-methyl-.beta.-phenyldiguanido-N5,N5')-hexane dihydrochloride; 1,6di(N₁,N₁'-p-nitrophenyldiguanido-N₅,N₅')hexane dihydrochloride;.omega.:.omega.'-di-(N1,N1'-phenyldiguanido-N5,N5')-di-n-propyl dihydrochloride;.omega:omega'-di(N1,N1'-p-chlorophenyldiguanidoether N₅,N₅')-di-n-propylether tetrahydrochloride; 1,6-di(N₁,N₁'-2,4-30 dichlorophenyldiguanido-N5,N5')hexane tetrahydrochloride; 1,6-di(N₁,N₁'-pmethylphenyldiguanido-N₅,N₅') hexane dihydrochloride; 1,6-di(N₁,N₁'-2,4,5trichlorophenyldiguanido-N5,N5') hexane tetrahydrochloride; 1,6-di[N₁,N₁'-.alpha.-(p-chlorophenyl) ethyldiguanido-N5,N5'] hexane dihydrochloride;.omega.:.omega.'di(N₁, N₁'-p-chlorophenyl diguanido-N₅,N₅')m-35 xylene dihydrochloride; 1,12-di(N₁,N₁'-p-chlorophenyl diguanido-N5,N5') dihydrochloride; 1,10-di(N₁,N₁'-phenyldiguanido-N₅,N₅')-decane dodecane

tetrahydrochloride; 1,12-di(N₁,N₁'-phenyldiguanido-N₅,N₅') dodecane tetrahydrochloride; 1,6-di(N₁,N₁'-o-chlorophenyldiguanido-N₅,N₅') hexane dihydrochloride; 1,6-di(N₁,N₁'-p-chlorophenyldiguanido-N₅,N₅')-hexane tetrahydrochloride; ethylene bis (1-tolyl biguanide); ethylene bis (p-tolyl 5 biguanide); ethylene bis(3,5-dimethylphenyl biguanide); ethylene bis(p-tertamylphenyl biguanide); ethylene bis(nonylphenyl biguanide); ethylene bis (phenyl biguanide); ethylene bis (N-butylphenyl biguanide); ethylene bis (2.5diethoxyphenyl biguanide); ethylene bis(2,4-dimethylphenyl biguanide); ethylene bis(o-diphenylbiguanide); ethylene bis(mixed amyl naphthyl biguanide); N-butyl 10 ethylene bis(phenylbiguanide); trimethylene bis(o-tolyl biguanide); N-butyl trimethylene bis(phenyl biguanide); and the corresponding pharmaceutically acceptable salts of all of the above such as the acetates; gluconates; hydrochlorides; hydrobromides; citrates; bisulfites; fluorides; polymaleates; Ncoconutalkylsarcosinates; phosphites; hypophosphites; perfluorooctanoates; 15 silicates; salicylates; sorbates; maleates; tartrates; fumarates; ethylenediaminetetraacetates; iminodiacetates: cinnamates; thiocyanates: pyromellitates: arginates: tetracarboxybutyrates: benzoates: glutarates: monofluorophosphates: and perfluoropropionates. mixtures and thereof. Preferred antimicrobials from this group are 1,6-di-(N₁,N₁'-phenyldiguanido-20 N₅,N₅')-hexane tetrahydrochloride: 1,6-di(N₁,N₁'-o-chlorophenyldiguanido-N₅,N₅')-hexane dihydrochloride; 1,6-di(N₁,N₁'-2,6-dichlorophenyldiguanido-N₅,N₅')hexane dihydrochloride; 1,6-di(N₁,N₁'-2,4-dichlorophenyldiguanido-N₅,N₅')hexane tetra hydrochloride; 1,6-di[N₁,N₁'-.alpha.-(p-chlorophenyl) ethyldiguanido-N5,N5'] hexane dihydrochloride;.omega.:.omega.'di(N1, N1'-p-25 chlorophenyldiguanido-N5,N5')m-xylene dihydrochloride; 1,12-di(N₁,N₁'-pchlorophenyldiguanido-N5,N5') dodecane dihydrochloride; 1,6-di(N₁,N₁'-ochlorophenyldiguanido-N5,N5') hexane dihydro chloride; 1,6-di(N₁,N₁'-pchlorophenyldiguanido-N5,N5')-hexane tetrahydrochloride; and mixtures thereof; 1,6-di(N₁,N₁'-o-chlorophenyldiguanido-N₅,N₅')-hexane more preferably, 30 dihydrochloride; 1,6-di(N₁,N₁'-2,6-dichlorophenyldiguanido-N₅,N₅') dihydrochloride; 1,6-di(N₁,N₁'-2,4-dichlorophenyldiguanido-N₅,N₅')hexane tetrahydrochloride; 1,6-di[N₁,N₁'-.alpha.-(p-chlorophenyl) ethyldiguanido-N₅,N₅'] hexane dihydrochloride;.omega.:.omega.'di(N1, N1'-p-chlorophenyldiguanido-N₅,N₅')m-xylene dihydrochloride; 1,12-di(N₁,N₁'-p-chlorophenyldiguanido-35 N5,N5') dodecane dihydrochloride; 1,6-di(N₁,N₁'-o-chlorophenyldiguanido-N₅,N₅') hexane dihydrochloride; 1,6-di(N₁,N₁'-p-chlorophenyldiguanido-N₅,N₅')- hexane tetrahydro chloride; and mixtures thereof. As stated hereinbefore, the bis biguanide of choice is chlorhexidine its salts, e.g., digluconate, dihydrochloride, diacetate, and mixtures thereof.

5 Quaternary Compounds. A wide range of quaternary compounds can also be used as antimicrobial actives, in conjunction with the preferred surfactants, for compositions of the present invention that do not contain cyclodextrin. Nonlimiting examples of useful quaternary compounds include: (1) benzalkonium chlorides and/or substituted benzalkonium chlorides such as commercially available Barquat® (available from Lonza), Maguat® (available from Mason), 10 Variquat® (available from Witco/Sherex), and Hyamine® (available from Lonza); (2) dialkyl guaternary such as Bardac® products of Lonza, (3) N-(3-chloroallyl) hexaminium chlorides such as Dowicide® and Dowicil® available from Dow; (4) benzethonium chloride such as Hyamine® 1622 from Rohm & Haas; (5) methylbenzethonium chloride represented by Hyamine® 10X supplied by Rohm 15 & Haas, (6) cetylpyridinium chloride such as Cepacol chloride available from of Typical concentrations for biocidal effectiveness of these quaternary compounds range from about 0.001% to about 0.8%, preferably from about 0.005% to about 0.3%, more preferably from about 0.01% to 0.2%, by 20 weight of the usage composition. The corresponding concentrations for the concentrated compositions are from about 0.003% to about 2%, preferably from about 0.006% to about 1.2%, and more preferably from about 0.1% to about 0.8% by weight of the concentrated compositions.

Other preservatives which are conventional in the art, such as described in US 5,593, 670 incorporated herein by reference, may also be used herein.

The surfactants, when added to the antimicrobials tend to provide improved antimicrobial action. This is especially true for the siloxane surfactants, and especially when the siloxane surfactants are combined with the chlorhexidine antimicrobial actives.

6. Polyolefin

30

35

The compositions of the present invention can also include an optional dispersible polyolefin. When used this will provide a further wrinkle reduction. Preferably, the polyolefin is a polyethylene, polypropylene or mixtures thereof.

10

15

20

25

30

35

The polyolefin may be at least partially modified to contain various functional groups, such as carboxyl, alkylamide, sulfonic acid or amide groups. More preferably, the polyolefin employed in the present invention is at least partially carboxyl modified or, in other words, oxidized. In particular, oxidized or carboxyl modified polyethylene is preferred in the compositions of the present invention.

For ease of formulation, the dispersible polyolefin is preferably introduced as a suspension or an emulsion of polyolefin dispersed by use of an emulsifying agent. The polyolefin suspension or emulsion preferably has from about 1 to about 50%, more preferably from about 10 to about 35% by weight, and most preferably from about 15 to about 30% by weight of polyolefin in the emulsion. The polyolefin preferably has a molecular weight of from about 500 to about 15,000 and more preferably from about 4,000 to about 10,000.

When an emulsion is employed, the emulsifier may be any suitable emulsification agent. Preferably, the emulsifier is a cationic or nonionic surfactant or mixtures thereof. Most any suitable cationic or nonionic surfactant may be employed as the emulsifier of the present invention. Preferred emulsifiers of the present invention are cationic surfactants such as the fatty amine surfactants and in particular the ethoxylated fatty amine surfactants. In particular, the cationic surfactants are preferred as emulsifiers in the present invention when the pH of the liquid composition is formulated in the preferred range of from about 2 to about 7. The dispersible polyolefin is dispersed by use of an emulsifier or suspending agent in a ratio of emulsifier to polyolefin of from about 1:10 to about Preferably, the emulsion includes from about 0.1 to about 50%, more preferably from about 1 to about 20% and most preferably from about 2.5 to about 10% by weight of emulsifier in the polyolefin emulsion. Polyethylene emulsions suitable for use in the present invention are available under the tradename VELUSTROL from HOECHST Aktiengesellschaft of Frankfurt am Main, Germany. In particular, the polyethylene emulsions sold under the tradename VELUSTROL PKS, VELUSTROL KPA and VELUSTROL P-40 may be employed in the compositions of the present invention.

The compositions of the present invention may contain from about 0.01% to about 50% by weight of the polyolefin. More preferably, the compositions include from about 0.1% to about 20% by weight polyolefin and most preferably from

about 0.1% to about 10% by weight polyolefin. When the dispersible polyolefin is added as an emulsion or suspension of polyolefin as described above, from about 0.1% to about 90%, and more preferably from about 0.5% to about 25% by weight of the emulsion or suspension may be added.

5

10

15

30

35

7. Hydroxy-capped polydimethyl siloxane emulsion (silanol)

When a polyolefin emulsion as described above under (6) is present in combination with a polymer such a polyvinyl alcohol, it has been found desirable to add a hydroxy-capped polydimethyl siloxane emulsion (silanol). Such addition will further increses the wrinkles reduction present on fabrics.

Typical of hydroxy-capped polydimethyl siloxane emulsion (silanol) also called Dimethiconols according to INCI and the CTFA, are SM2725 and SM2068A commercially available from General Electric, Q2-1403 from Dow Corning; Abil OSW series from Goldschmidt; and Sandoperm FE from Sandoz. A preferred silanol for use herein, especially when used in combination with a polyvinylalcohol and polyolefin emulsion is SM2725 because of its film forming properties.

The compositions of the present invention may contain from about 0.01% to about 25% by weight of the hydroxy-capped polydimethyl siloxane emulsion. More preferably, the compositions include from about 0.01% to about 20% by weight hydroxy-capped polydimethyl siloxane emulsion and most preferably from about 0.1% to about 10% by weight hydroxy-capped polydimethyl siloxane emulsion.

8- Perfume

The composition of the present invention can also optionally provide a "scent signal" in the form of a pleasant odor which signals the removal of malodor from fabrics. The scent signal is designed to provide a fleeting perfume scent, and is not designed to be overwhelming or to be used as an odor masking ingredient. When perfume is added as a scent signal, it is added only at very low levels, e.g., from about 0% to about 0.5%, preferably from about 0.003% to about 0.3%, more preferably from about 0.005% to about 0.2%, by weight of the usage composition.

Perfume can also be added as a more intense odor in product and on surfaces. When stronger levels of perfume are preferred, relatively higher levels of perfume can be added. Any type of perfume can be incorporated into the composition of the present invention.

5

10

20

25

Preferably the perfume is hydrophilic and is composed predominantly of ingredients selected from two groups of ingredients, namely, (a) hydrophilic ingredients having a ClogP of less than about 3.5, more preferably less than about 3.0, and (b) ingredients having significant low detection threshold, and mixtures thereof. Typically, at least about 50%, preferably at least about 60%, more preferably at least about 70%, and most preferably at least about 80% by weight of the perfume is composed of perfume ingredients of the above groups (a) and (b).

15 (a). <u>Hydrophilic Perfume Ingredients</u>

The hydrophilic perfume ingredients are more soluble in water, have less of a tendency to complex with the cyclodextrins, and are more available in the odor absorbing composition than the ingredients of conventional perfumes. The degree of hydrophobicity of a perfume ingredient can be correlated with its octanol/water partition coefficient P. The octanol/water partition coefficient of a perfume ingredient is the ratio between its equilibrium concentration in octanol and in water. A perfume ingredient with a greater partition coefficient P is considered to be more hydrophobic. Conversely, a perfume ingredient with a smaller partition coefficient P is considered to be more hydrophilic. Since the partition coefficients of the perfume ingredients normally have high values, they are more conveniently given in the form of their logarithm to the base 10, logP. Thus the preferred perfume hydrophilic perfume ingredients of this invention have logP of about 3.5 or smaller, preferably of about 3.0 or smaller.

The logP of many perfume ingredients have been reported; for example, the Pomona92 database, available from Daylight Chemical Information Systems, Inc. (Daylight CIS), Irvine, California, contains many, along with citations to the original literature. However, the logP values are most conveniently calculated by the "CLOGP" program, also available from Daylight CIS. This program also lists experimental logP values when they are available in the Pomona92 database. The "calculated logP" (ClogP) is determined by the fragment approach of Hansch

and Leo (cf., A. Leo, in Comprehensive Medicinal Chemistry, Vol. 4, C. Hansch, P. G. Sammens, J. B. Taylor and C. A. Ramsden, Eds., p. 295, Pergamon Press, 1990, incorporated herein by reference). The fragment approach is based on the chemical structure of each perfume ingredient, and takes into account the numbers and types of atoms, the atom connectivity, and chemical bonding. The ClogP values, which are the most reliable and widely used estimates for this physicochemical property, are used instead of the experimental logP values in the selection of perfume ingredients which are useful in the present invention.

5

10

15

20

25

Non-limiting examples of the more preferred hydrophilic perfume ingredients are allyl amyl glycolate, allyl caproate, amyl acetate, amyl propionate, anisic aldehyde, anisyl acetate, anisole, benzaldehyde, benzyl acetate, benzyl acetone, benzyl alcohol, benzyl formate, benzyl iso valerate, benzyl propionate, beta gamma hexenol, calone, camphor gum, laevo-carveol, d-carvone, laevo-carvone, cinnamic alcohol, cinnamyl acetate, cinnamic alcohol, cinnamyl formate, cinnamyl propionate, cis-jasmone, cis-3-hexenyl acetate, coumarin, cuminic alcohol, cuminic aldehyde, Cyclal C, cyclogalbanate, dihydroeuginol, dihydro isojasmonate, dimethyl benzyl carbinol, dimethyl benzyl carbinyl acetate, ethyl acetate, ethyl aceto acetate, ethyl amyl ketone, ethyl anthranilate, ethyl benzoate, ethyl butyrate, ethyl cinnamate, ethyl hexyl ketone, ethyl maltol, ethyl-2-methyl butyrate, ethyl methylphenyl glycidate, ethyl phenyl acetate, ethyl salicylate, ethyl vanillin, eucalyptol, eugenol, eugenyl acetate, eugenyl formate, eugenyl methyl ether, fenchyl alcohol, flor acetate (tricyclo decenyl acetate), geranyl geraniol, propionate), decenvl (tricyclo fructone, frutene oxyacetaldehyde, heliotropin, hexenol, hexenyl acetate, hexyl formate, hinokitiol, hydratropic alcohol, hydroxycitronellal, hydroxycitronellal diethyl acetal, hydroxycitronellol, indole, isoamyl alcohol, iso cyclo citral, isoeugenol, isoeugenyl acetate, isomenthone, isopulegyl acetate, isoquinoline, keone, ligustral, linalool, linalool oxide, linalyl formate, lyral, menthone, methyl acetophenone, methyl amyl ketone, methyl anthranilate, methyl benzoate, methyl 30 benzyl acetate, methyl cinnamate, methyl dihydrojasmonate, methyl eugenol, methyl heptenone, methyl heptine carbonate, methyl heptyl ketone, methyl hexyl ketone, methyl isobutenyl tetrahydropyran, methyl-N-methyl anthranilate, methyl beta naphthyl ketone, methyl phenyl carbinyl acetate, methyl salicylate, nerol, nonalactone, octalactone, octyl alcohol (octanol-2), para-anisic aldehyde, para-35 cresol, para-cresyl methyl ether, para hydroxy phenyl butanone, para-methoxy

10

15

20

25

acetophenone, para-methyl acetophenone, phenoxy ethanol, phenoxyethyl propionate, phenyl acetaldehyde, phenylacetaldehyde diethyl ether, phenylethyl oxyacetaldehyde, phenyl ethyl acetate, phenyl ethyl alcohol, phenyl ethyl dimethyl carbinol, prenyl acetate, propyl butyrate, pulegone, rose oxide, safrole, terpineol, vanillin, viridine, and mixtures thereof.

Nonlimiting examples of other preferred hydrophilic perfume ingredients which can be used in perfume compositions of this invention are allyl heptoate, amyl benzoate, anethole, benzophenone, carvacrol, citral, citronellol, citronellyl nitrile, cyclohexyl ethyl acetate, cymal, 4-decenal, dihydro isojasmonate, dihydro myrcenol, ethyl methyl phenyl glycidate, fenchyl acetate, florhydral, gammanonalactone, geranyl formate, geranyl nitrile, hexenyl isobutyrate, alpha-ionone, isobornyl acetate, isobutyl benzoate, isononyl alcohol, isomenthol, paraisopropyl phenylacetaldehyde, isopulegol, linaly acetate, 2-methoxy naphthalene, menthyl acetate, methyl chavicol, musk ketone, beta naphthol methyl ether, neral, nonyl aldehyde, phenyl heptanol, phenyl hexanol, terpinyl acetate, Veratrol, yara-yara, and mixtures thereof.

The preferred perfume compositions used in the present invention contain at least 4 different hydrophilic perfume ingredients, preferably at least 5 different hydrophilic perfume ingredients, more preferably at least 6 different hydrophilic perfume ingredients, and even more preferably at least 7 different hydrophilic perfume ingredients. Most common perfume ingredients which are derived from natural sources are composed of a multitude of components. When each such material is used in the formulation of the preferred perfume compositions of the present invention, it is counted as one single ingredient, for the purpose of defining the invention.

(b). Low Odor Detection Threshold Perfume Ingredient

The odor detection threshold of an odorous material is the lowest vapor concentration of that material which can be olfactorily detected. The odor detection threshold and some odor detection threshold values are discussed in, e.g., "Standardized Human Olfactory Thresholds", M. Devos et al, IRL Press at Oxford University Press, 1990, and "Compilation of Odor and Taste Threshold Values Data", F. A. Fazzalari, editor, ASTM Data Series DS 48A, American Society for Testing and Materials, 1978, both of said publications being

10

15

20

incorporated by reference. The use of small amounts of perfume ingredients that have low odor detection threshold values can improve perfume odor character, even though they are not as hydrophilic as perfume ingredients of group (a) which are given hereinabove. Perfume ingredients that do not belong to group (a) above, but have a significantly low detection threshold, useful in the composition of the present invention, are selected from the group consisting of ambrox, bacdanol, benzyl salicylate, butyl anthranilate, cetalox, damascenone, alpha-damascone, gamma-dodecalactone, ebanol, herbavert, cis-3-hexenyl salicylate, alpha-ionone, beta-ionone, alpha-isomethylionone, lilial, methyl nonyl ketone, gamma-undecalactone, undecylenic aldehyde, and mixtures thereof. These materials are preferably present at low levels in addition to the hydrophilic ingredients of group (a), typically less than about 20%, preferably less than about 15%, more preferably less than about 10%, by weight of the total perfume compositions of the present invention. However, only low levels are required to provide an effect.

There are also hydrophilic ingredients of group (a) that have a significantly low detection threshold, and are especially useful in the composition of the present invention. Examples of these ingredients are allyl amyl glycolate, anethole, benzyl acetone, calone, cinnamic alcohol, coumarin, cyclogalbanate, Cyclal C, cymal, 4-decenal, dihydro isojasmonate, ethyl anthranilate, ethyl-2-methyl butyrate, ethyl methylphenyl glycidate, ethyl vanillin, eugenol, flor acetate, florhydral, fructone, frutene, heliotropin, keone, indole, iso cyclo citral, isoeugenol, lyral, methyl heptine carbonate, linalool, methyl anthranilate, methyl dihydrojasmonate, methyl isobutenyl tetrahydropyran, methyl beta naphthyl ketone, beta naphthol methyl ether, nerol, para-anisic aldehyde, para hydroxy phenyl butanone, phenyl acetaldehyde, vanillin, and mixtures thereof. Use of low odor detection threshold perfume ingredients minimizes the level of organic material that is released into the atmosphere.

30

35

25

9-Salt to absorb amine and sulfur-containing compounds

Optionally, but preferably, salts to absorb amine and sulfur-containing compounds, preferably water-soluble zinc salts, can be added to the composition of the present invention. The salt can be used as an odor control agent. The water-soluble metallic salt can be present in the composition of the present invention to absorb amine and sulfur-containing compounds. Furthermore, they

usually do not contribute an odor of their own. Preferably the water-soluble metallic salts are selected from the group consisting of copper salts, zinc salts, and mixtures thereof.

Metallic salts of use herein are described in US 5670475 column 9 line 8 to 43. Examples of preferred water-soluble zinc salts are zinc chloride, zinc gluconate, zinc lactate, zinc maleate, zinc salicylate, zinc sulfate, etc. Highly-ionized and soluble zinc salts such as zinc chloride, provide the best source of zinc ions. Examples of preferred copper salts are copper chloride and copper gluconate.

10 Preferred metallic salts are zinc chloride and copper chloride.

Salts to absorb amine and sulfur-containing compounds are added to the composition of the present invention typically at a level of from about 0.1% to about 10%, preferably from about 0.2% to about 7%, more preferably from about 0.3% to about 5%, by weight of the composition.

10-Soil Release Agent

15

20

25

35

Soil Release agents are desirably used in compositions of the instant invention. Any polymeric soil release agent known to those skilled in the art can optionally be employed in the compositions of this invention. Polymeric soil release agents are characterized by having both hydrophilic segments, to hydrophilize the surface of hydrophobic fibers, such as polyester and nylon, and hydrophobic segments, to deposit upon hydrophobic fibers and remain adhered thereto through completion of washing and rinsing cycles and, thus, serve as an anchor for the hydrophilic segments. This can enable stains occurring subsequent to treatment with the soil release agent to be more easily cleaned in later washing procedures.

If utilized, soil release agents will generally comprise from about 0.01% to about 10.0%, by weight, of the compositions herein, typically from about 0.1% to about 5%, preferably from about 0.2% to about 3.0%.

The following, all included herein by reference, describe soil release polymers suitable for use in the present invention. U.S. 3,959,230 Hays, issued May 25, 1976; U.S. 3,893,929 Basadur, issued July 8, 1975; U.S. 4,000,093, Nicol, *et al.*, issued December 28, 1976; U.S. Patent 4,702,857 Gosselink, issued October

27, 1987; U.S. 4,968,451, Scheibel *et al.*, issued November 6; U.S. 4,702,857, Gosselink, issued October 27, 1987; U.S. 4,711,730, Gosselink *et al.*, issued December 8, 1987; U.S. 4,721,580, Gosselink, issued January 26, 1988; U.S. 4,877,896, Maldonado *et al.*, issued October 31, 1989; U.S. 4,956,447, Gosselink *et al.*, issued September 11, 1990; U.S. 5,415,807 Gosselink *et al.*, issued May 16, 1995; European Patent Application 0 219 048, published April 22, 1987 by Kud, *et al.*.

Further suitable soil release agents are described in U.S. 4,201,824, Violland et al.; U.S. 4,240,918 Lagasse et al.; U.S. 4,525,524 Tung et al.; U.S. 4,579,681, Ruppert et al.; U.S. 4,240,918; U.S. 4,787,989; U.S. 4,525,524; EP 279,134 A, 1988, to Rhone-Poulenc Chemie; EP 457,205. A to BASF (1991); and DE 2,335,044 to Unilever N. V., 1974 all incorporated herein by reference.

15 Commercially available soil release agents include the METOLOSE SM100, METOLOSE SM200 manufactured by Shin-etsu Kagaku Kogyo K.K., SOKALAN type of material, e.g., SOKALAN HP-22, available from BASF (Germany), ZELCON 5126 (from Dupont) and MILEASE T (from ICI).

20 11-Pro-perfume

The composition may also comprises ingredient useful for providing a long and lasting release of a perfume material. Typical disclosure can be found in WO95/04809, WO96/02625, PCT US97/14610 filed 19 August 1997 and claiming priority of 19 August 1996, and EP-A-0,752,465.

Typical level of incorporation of the perfume are from 0.01% to 15% by weight of the composition.

12-pH

30

An optional requirement of the compositions according to the present invention is that the pH is greater than 7, preferably between 7 and 12, more preferably between 8 and 11, most preferably between 9 and 10.5. This is achieved by the addition of a caustic alkali. Suitable caustic alkalis for use herein include sodium and potassium hydroxide.

35 13- Other Optional Ingredients

10

15

20

25

30

35

The present invention can include optional components conventionally used in textile treatment compositions, for example, colorants, preservatives, bactericides, optical brighteners, opacifiers, anti-shrinkage agents, germicides, fungicides, anti-oxidants, dye fixing agent, enzymes, chelating agents, color protectors like polyethylene imine and its alkoxylated derivatives, and the like. The compositions are preferably free of any material that would soil or stain fabric, and are also substantially free of starch. Typically, there should be less than about 0.5%, by weight of the composition, preferably less than about 0.3%, more preferably less than about 0.1%, by weight of the composition, of starch and/or modified starch.

E. Form of the composition and compounds

The composition or its individual components can be provided in any suitable form such as spray, foam, gel or any other suitable form for liquid aqueous compositions, preferably the composition is in the form of a spray. Preferably, when sprayed, the liquid composition which is applied on the fabric will have particle sizes in the range of 8 to 100 μm , preferably from 10-60 μm (more preferably from 20-60 μm) for automatic sprayer, and preferably from 50-100 μm for manually activated sprayer. Accordingly, there is provided a packaged composition comprising the composition or compounds, preferably in a spray dispenser.

F. Packaging

In another aspect of the invention, a packaged composition is provided that comprises a packaged composition comprising a wrinkle reducing composition comprising a wrinkle reducing active which contains a polymer, a water-solubilising agent, a liquid carrier, and a spray dispensing device.

The dilute compositions, i.e., compositions containing from about 0.1% to about 5%, by weight of the composition, of wrinkle reducing active, of the present invention are preferably sprayed onto fabrics and therefore are typically packaged in a spray dispenser. The spray dispenser can be any of the manually activated means for producing a spray of liquid droplets as is known in the art, e.g. trigger-type, pump-type, electrical spray, hydraulic nozzle, sonic nebulizer, high pressure fog nozzle, non-aerosol self-pressurized, and aerosol-type spray means. Automatic activated means can also be used herein. These type of

WO 00/08249

automatic means are similar to manually activated means with the exception that the propellant is replaced by a compressor. It is preferred that at least about 70%, more preferably, at least about 80%, most preferably at least about 90% of the droplets have a particle size of smaller than about 200 microns.

5

10

15

20

25

The spray dispenser can be an aerosol dispenser. Said aerosol dispenser comprises a container which can be constructed of any of the conventional materials employed in fabricating aerosol containers. The dispenser must be capable of withstanding internal pressure in the range of from about 5 to about 100 p.s.i.g., more preferably from about 10 to about 60 p.s.i.g. The one important requirement concerning the dispenser is that it be provided with a valve member which will permit the wrinkle reducing composition contained in the dispenser to be dispensed in the form of a spray of very fine, or finely divided, particles or droplets. The aerosol dispenser utilizes a pressurized sealed container from which the wrinkle reducing composition is dispensed through a special actuator/valve assembly under pressure. The aerosol dispenser is pressurized by incorporating therein a gaseous component generally known as a propellant. Common aerosol propellants, e.g., gaseous hydrocarbons such as isobutane, and mixed halogenated hydrocarbons, are not preferred. Halogenated hydrocarbon propellants such as chlorofluoro hydrocarbons have been alleged to contribute to environmental problems. Preferred propellants are compressed air, nitrogen, inert gases, carbon dioxide, etc. A more complete description of commercially available aerosol-spray dispensers appears in U.S. Pat. Nos.: 3,436,772, Stebbins, issued April 8, 1969; and 3,600,325, Kaufman et al., issued August 17, 1971; both of said references are incorporated herein by reference.

30

35

Preferably the spray dispenser can be a self-pressurized non-aerosol container having a convoluted liner and an elastomeric sleeve. Said self-pressurized dispenser comprises a liner/sleeve assembly containing a thin, flexible radially expandable convoluted plastic liner of from about 0.010 to about 0.020 inch thick, inside an essentially cylindrical elastomeric sleeve. The liner/sleeve is capable of holding a substantial quantity of odor-absorbing fluid product and of causing said product to be dispensed. A more complete description of self-pressurized spray dispensers can be found in U.S. Pat. Nos. 5,111,971, Winer, issued May 12, 1992, and 5,232,126, Winer, issued Aug. 3, 1993; both of said references are herein incorporated by reference. Another type of aerosol spray dispenser is one

wherein a barrier separates the wrinkle reducing composition from the propellant (preferably compressed air or nitrogen), as is disclosed in U.S. Pat. No. 4,260,110, issued April 7, 1981, incorporated herein by reference. Such a dispenser is available from EP Spray Systems, East Hanover, New Jersey.

5

10

15

20

25

30

35

More preferably, the spray dispenser is a non-aerosol, manually activated, pump-spray dispenser. Said pump-spray dispenser comprises a container and a pump mechanism which securely screws or snaps onto the container. The container comprises a vessel for containing the wrinkle reducing composition to be dispensed.

The pump mechanism comprises a pump chamber of substantially fixed volume, having an opening at the inner end thereof. Within the pump chamber is located a pump stem having a piston on the end thereof disposed for reciprocal motion in the pump chamber. The pump stem has a passageway there through with a dispensing outlet at the outer end of the passageway and an axial inlet port located inwardly thereof.

The container and the pump mechanism can be constructed of any conventional material employed in fabricating pump-spray dispensers, including, but not limited to: polyethylene; polypropylene; polyethyleneterephthalate; blends of polyethylene, vinyl acetate, and rubber elastomer. Other materials can include stainless steel. A more complete disclosure of commercially available dispensing devices appears in: U.S. Pat. Nos.: 4,895,279, Schultz, issued January 23, 1990; 4,735,347, Schultz et al., issued April 5, 1988; and 4,274,560, Carter, issued June 23, 1981; all of said references are herein incorporated by reference.

Most preferably, the spray dispenser is a manually activated trigger-spray dispenser. Said trigger-spray dispenser comprises a container and a trigger both of which can be constructed of any of the conventional material employed in fabricating trigger-spray dispensers, including, but not limited to: polyethylene; polypropylene; polyacetal; polycarbonate; polyethyleneterephthalate; polyvinyl chloride; polystyrene; blends of polyethylene, vinyl acetate, and rubber elastomer. Other materials can include stainless steel and glass. The trigger-spray dispenser does not incorporate a propellant gas. The trigger-spray dispenser herein is typically one which acts upon a discrete amount of the

wrinkle reducing composition itself, typically by means of a piston or a collapsing bellows that displaces the composition through a nozzle to create a spray of thin liquid. Said trigger-spray dispenser typically comprises a pump chamber having either a piston or bellows which is movable through a limited stroke response to the trigger for varying the volume of said pump chamber. This pump chamber or bellows chamber collects and holds the product for dispensing. The trigger spray dispenser typically has an outlet check valve for blocking communication and flow of fluid through the nozzle and is responsive to the pressure inside the chamber. For the piston type trigger sprayers, as the trigger is compressed, it acts on the fluid in the chamber and the spring, increasing the pressure on the For the bellows spray dispenser, as the bellows is compressed, the pressure increases on the fluid. The increase in fluid pressure in either trigger spray dispenser acts to open the top outlet check valve. The top valve allows the product to be forced through the swirl chamber and out the nozzle to form a discharge pattern. An adjustable nozzle cap can be used to vary the pattern of the fluid dispensed.

For the piston spray dispenser, as the trigger is released, the spring acts on the piston to return it to its original position. For the bellows spray dispenser, the bellows acts as the spring to return to its original position. This action causes a vacuum in the chamber. The responding fluid acts to close the outlet valve while opening the inlet valve drawing product up to the chamber from the reservoir.

A more complete disclosure of commercially available dispensing devices appears in U.S. Pat. Nos. 4,082,223, Nozawa, issued Apr. 4, 1978; 4,161, 288, McKinney, issued Jul. 17, 1985; 4,434,917, Saito et al., issued Mar. 6, 1984; and 4,819,835, Tasaki, issued Apr. 11, 1989; 5,303,867, Peterson, issued Apr. 19, 1994; all of said references are incorporated herein by reference.

A broad array of trigger sprayers or finger pump sprayers are suitable for use with the compositions of this invention. These are readily available from suppliers such as Calmar, Inc., City of Industry, California; CSI (Continental Sprayers, Inc.), St. Peters, Missouri; Berry Plastics Corp., Evansville, Indiana - a distributor of Guala® sprayers; or Seaquest Dispensing, Cary, Illinois.

5

10

15

20

The preferred trigger sprayers are the blue inserted Guala® sprayer, available from Berry Plastics Corp., the Calmar TS800-1A® sprayers, available from Calmar Inc., or the CSI T7500® available from Continental Sprayers, Inc., because of the fine uniform spray characteristics, spray volume, and pattern size. Any suitable bottle or container can be used with the trigger sprayer, the preferred bottle is a 17 fl-oz. bottle (about 500 ml) of good ergonomics similar in shape to the Cinch® bottle. It can be made of any materials such as high density polyethylene, polypropylene, polyvinyl chloride, polystyrene, polyethylene terephthalate, glass, or any other material that forms bottles. Preferably, it is made of high density polyethylene or polyethylene terephthalate.

For smaller four fl-oz. size (about 118 ml), a finger pump can be used with canister or cylindrical bottle. The preferred pump for this application is the cylindrical Euromist II® from Seaguest Dispensing.

15

20

25

35

10

5

Regardless of the particular commercial spray nozzle used, it is preferable for the atomization spray nozzle to have an orifice diameter of from about 0.1 mm to about 2 mm, and most preferably from about 0.15 mm to about 1 mm. The spraying step is conducted for a period of time of from about 5 minutes to about 30 minutes, more preferably from about 5 minutes to about 20 minutes. Spraying times will vary depending upon the various operating parameters selected as described herein.

For use herein, it is preferred that said spray dispenser comprises a trigger spray device. More preferably, the spray dispenser should be capable of providing droplets with a weight average diameter of from 8 to 100 μ m, preferably from 10-60 μ m (more preferably from 20-60 μ m) for automatic sprayer, and preferably from 50-100 μ m for manually activated sprayer.

30 G. Method of use

An effective amount of the composition of the present invention is preferably sprayed onto fabrics, particularly clothing. When the composition is sprayed onto fabric an effective amount should be deposited onto the fabric without causing saturation of the fabric, typically from 3 % to 85%, preferably from 5 % to 50%, more preferably from 5 % to 25 %, by weight of the fabric. The amount of total active typically sprayed onto the fabric is from 0.01% to 3 %, preferably from

15

20

25

30

0.1% to 2 %, more preferably from 0.1 % to 1 %, by weight of the fabric. Once an effective amount of the composition is sprayed onto the fabric the fabric is optionally, but preferably stretched. The fabric is typically stretched perpendicular to the wrinkle. The fabric can also be smoothed by hand after it has been sprayed. The smoothing movement works particularly well on areas of clothing that have interface sewn into them, or on the hem of clothing. Once the fabric has been sprayed and optionally, but preferably, stretched, it is hung until dry.

10 Accordingly, there is provided a method for reducing wrinkles on fabrics which comprises the steps of contacting the fabrics with a composition of the invention, as defined herein before.

By "contacting", it is meant any steps that is suitable for providing a contact of the composition with the fabric. This can include by soaking, washing, rinsing, and/or spraying as well as by means of a dryer sheet onto which is adsorbed the composition.

Further, the use of a water-soluble surfactant has surprisingly been found to solubilise the polymer of the invention composition. Typical of these surfactants are the surfactant herein before described under solubilising agents as well as cationic surfactants such as choline ester surfactants, alkyl imidazolinium salts, or mixtures thereof as described in WO 93/23510 as single long chain alkyl cationic surfactants. Accordingly, there is also provided a process for solubilising a polymer as defined herein before by means of a solubilising agent selected from water-soluble surfactants and mixtures thereof.

The composition of the present invention can also be used as an ironing aid. An effective amount of the composition can be sprayed onto fabric, wherein said fabric should not be sprayed to saturation. The fabric can be ironed at the normal temperature at which it should be ironed. The fabric can be sprayed with an effective amount of the composition, allowed to dry and then ironed, or sprayed and ironed immediately.

In a still further aspect of the invention, the composition can be sprayed onto fabrics by means of an in-home de-wrinkling apparatus containing the fabric to

10

15

20

25

30

35

be dewrinkled, thereby providing ease of operation. Conventional personal as well as industrial de-wrinkling apparatus are suitable for use herein. Traditionally, these apparatus act by a steaming process which provides a relaxing of the fibers. The spraying of the composition or compounds on the fabrics can then occurs within the chamber of the apparatus or before placing the fabrics into the chamber. Again, the spraying means should preferably be capable of providing droplets with a mean diameter of from 3 to 50 µm, preferably from 5-30 µm for automatic sprayer, and preferably from 50-100 µm for manually activated sprayer. Preferably, the loading of moisture on fabrics made of natural and synthetic fibers is from 5 to 25%, more preferably from 5 to 10% by weight of the dried fabric. Other conventional steps for the dewrinkling apparatus can be applied such as heating and drying. In one mode of operation, the liquid conditioning composition is fogged by combining it with an air stream under pressure and passed through the atomization nozzle in the cabinet. Preferably, for optimum dewrinkling benefit, the of the air stream and/or the conditioning composition can be heated to enhance distribution and deposition of the conditioning composition on the garments. In that regard, the temperature of the air stream and/or the conditioning composition can be as low as room temperature, and preferably is from 35C to 80, more preferably from 40-70C. By having the air stream and/or the conditioning composition at the aforementioned elevated temperatures, it has been found that superior de-wrinkling benefits are It should be understood that the temperature of the conditioning composition can be from about ambient (15°C) temperature to about 80°C, and higher temperatures generally improve de-wrinkling performance. The preferred length of the drying cycle is from 20 to 60 minutes, more preferably 30 to 45 minutes.

It has also been found that effective softening composition distribution on the garments, e.g inside the cabinet, can be further enhanced by optimally selecting the fluid surface tension of the softening composition. For example, it is preferable for the softening composition to have fluid surface tension of from about 5 dynes/cm to about 60 dynes/cm, more preferably of from about 20 dynes/cm to about 55 dynes/cm, and most preferably, from about 20 dynes/cm to about 30 dynes/cm. The lower surface tension of the softening composition improves effective distribution by improving surface absorption and spreading of the softening composition on the garment fabric.

15

20

30

Furthermore, it is preferable for the softening composition to have a fluid viscosity of from about 1 cps to about 100 cps, more preferably from about 1 cps to about 50 cps, and most preferably of from about 1 cps to about 20 cps as measured by a standard Brookfield viscometer.

In the examples, the abbreviated component identifications have the following meanings:

Solubilising agent 1: Linear secondary alcohol with 12 to 14 carbon atoms per molecules and 9 moles of ethylene oxide per mole equivalent of alcohol, commercially available under the tradename Softanol 90 and marketed by BP Chemicals Ltd.

Solubilising agent 2: branched chain alcohol ethylene oxide condensate having 6 moles of ethylene oxide per mole of alcohol commercially available under the tradename Lial 111 EO 6 and marketed by Enichem

Solubilising agent 3: Linear primary alcohol with 12 to 14 carbon atoms per molecules and 7 moles of ethylene oxide per mole equivalent of alcohol commercially available under the tradename Marlipal 24/70 and marketed by Huls

Solubilising agent 4: Silwet L-7600 commercially available from OSi Specialties

Lubricant: N,N-di-(canolyl-oxy-ethyl)-N-methyl-N-(2-hydroxyethyl) ammonium methyl sulfate

25 Cyclodextrin: Hydroxypropyl beta-cyclodextrin

preservative: Kathon

Dye fixative: Cationic dye fixing agent (50% active) available under the

tradename Tinofix Eco from Ciba-Geigy

Carezyme: Cellulytic enzyme of activity 1000 CEVU/g sold by NOVO

Industries A/S and of activity mentioned above unless

otherwise specified

TABLE 1

The following table defines the silicone copolymers used in the examples.

Synthesis of these compounds is made according the synthesis of Poly[(t-butyl acrylate)-graft-polydimethylsiloxane)] described in EP 412 704 and WO97/34572

or synthesis of tert-butyl acrylate/acrylic acid/ polydimethylsiloxane described in WO 97/15275.

5	Copolymer #1	10/70/20 acrylic acid/n-butylmethacrylate/silicone macromer S2, copolymer of molecular weight about 100,000			
	Copolymer #2	10/70/20 dimethylacrylamide/isobutyl methacrylate/silicone macromer S2, copolymer of molecular weight about 400,000			
0	Copolymer #3	60/20/20 diallyldimethyl ammonium chloride/isobutyl methacrylate/silicone macromer S1, copolymer of molecular weight about 500,000			
5	Copolymer #4	40/40/20 acrylic acid/methyl methacrylate/silicone macromer S1, copolymer of molecular weight about 400,000			
)	Copolymer #5	63/20/17 tert-butyl acrylate/acrylic acid/ polysiloxane- containing C momomer S1 polymer molecular weigh between about 120,000 and 150,000			
5	in a manner simi issued Mar. 1, 19 10,000 and is pre	r S1- has a molecular weight of about 15,000 and is prepared lar to Example C-2c of U.S. Pat. No. 4,728,571, Clemens 188. Silicone macromer S2- has a molecular weight of about pared in a manner similar to Example C-2b of U.S. Pat. No. 1, 1988.			
ס	TABLE 2 The following table defines other adhesive copolymers used in the example Synthesis of these compounds is made according the synthesis of US 5,120,53				
c	Copolymer #6	20/80 by weight of acrylic acid/ter-butyl acrylate of molecula weight is about 80,000 to 110,000.			
35					

WO 00/08249 PCT/US98/16127

84

	Copolymer #7	ethyl acrylate/metacrylic acid/methyl methacrylate/acrylic acid copolymer commercially available from Union Carbide under the tradename Amerhold DR-25
5	Copolymer #8	polyvinyl acetate commercially available from Clariant under the tradename APPRETAN
10	Copolymer #9	polyacrylate dispersion commercially available from BASF under the tradename Poligen A
.•	Copolymer #10	polyvinyl alcohol commercially available from Clariant under the tradename MOWEOL
15	Copolymer #11	polyvinylpyrrolidone/acrylic acid commercially available from BASF under the tradename SOKALAN EG 310

TABLE 3

The following table defines silicone adhesive block polymers used in the examples synthesized as followed:

20

25

30

35

Polymer #12: [Poly(dimethyl siloxane)-block-poly(t-butylacrylate-co-acrylic acid)]_n,

Place 62.4 parts of t-butyl acrylate, 20.8 parts acrylic acid, 200 parts acetone (as a solvent), and 15 parts isopropanol (as a chain transfer agent) in a roundbottom flask. Separately, dissolve 16.8 parts VPS-1001(poly(dimethyl siloxane)initiator) macroazoinitiator (commercially available from Wako Chemical USA, INC., Richmond, VA) into 200 parts ethyl acetate and add the solution to an addition funnel. Purge the reaction vessel with argon for approximately one hour. Following the purge, maintain a constant positive pressure on the closed reaction system with argon. Heat the reaction mixture to 58°C. Add the VPS-1001 solution to the reaction flask in a dropwise fashion over the course of one hour. Maintain heating and stirring for 20 hours. Terminate the reaction by opening the reactor to the atmosphere and cooling to room temperature.

The block copolymer is precipitated from the solution by adding one part of the polymer solution to 15 parts water. The resultant polymer is redissolved in acetone and is precipitated again from 15 parts water. The resultant block

copolymer is then placed in a vacuum oven for heated drying. Following the drying, the polymer is ground and extracted for 20 hours with hexane using a soxhlet extractor. The polymer is then vacuum dried with heat in an oven. The glass transition temperature of the block B of the resulting polymer is between about -20°C and 60°C.

Polymer #13 [Poly(dimethyl siloxane)-block-poly(t-butyl acrylate-co-n-butyl acrylate-co-acrylic acid-co-methacrylic acid)]n

Place 80 parts of t-butyl acrylate, 56 parts n-butyl acrylate, 12 parts acrylic acid, 10 parts methacrylic acid, 400 parts acetone (as a solvent), and 50 parts isopropanol (as a chain transfer agent) in a roundbottom flask. Separately, dissolve 20 parts VPS-1001(poly(dimethyl siloxane)initiator) macroazoinitiator (commercially available from Wako Chemical USA, INC., Richmond, VA) into 400 parts ethyl acetate, add to this solution 12 parts acrylic acid and 10 parts methacrylic acid, and add the solution to an addition funnel. Purge the reaction vessel with argon for approximately one hour. Following the purge, maintain a constant positive pressure on the closed reaction system with argon. Heat the reaction mixture to 58°C. Add the VPS-1001 solution to the reaction flask in a dropwise fashion over the course of one hour. Maintain heating and stirring for 20 hours. Terminate the reaction by opening the reactor to the atmosphere and cooling to room temperature.

The block copolymer is precipitated from the solution by adding one part of the polymer solution to 15 parts water. The resultant polymer is redissolved in acetone and is precipitated again from 15 parts water. The resultant block copolymer is then placed in a vacuum oven for heated drying. Following the drying, the polymer is ground and extracted for 20 hours with hexane using a soxhlet extractor. The polymer is then vacuum dried with heat in an oven. The glass transition temperature of the block B of the resulting polymer is between about -20°C and 60°C.

The invention is illustrated in the following non limiting examples, in which all percentages are on a weight basis unless otherwise stated.

5

10

15

20

25

30

Example I

The following compositions are in accordance with the present invention.

10

	A	В	С	D	E	F
Co-polymer #1	0.75%	-	-	-	-	-
Co-polymer #2	-	0.75%	-	-	-	-
Co-polymer #3	-	-	1.50%	-	-	-
Co-polymer #4	-	-	-	1.50%	-	
Co-polymer #5	_	-	-	-	0.50%	-
Co-polymer #6	-	-	-	-	-	0.50%
water-solubilising	0.50%	-	-	1.50%	-	-
agent 1						
water-solubilising	-	0.50%	-	-	0.50%	-
agent 2						0.500/
water-solubilising	-	-	1.50%	-	-	0.50%
agent 3					0.050/	0.25%
water-solubilising	0.25%	0.25%	0.25%	0.25%	0.25%	0.25%
agent 4					0.500/	0.50%
Dipropyleneglycol	-	-	-	-	0.50%	0.50%
Diethyleneglycol	0.75%	0.75%	0.75%	0.75%	-	
Cyclodextrin	1.00%	1.00%	1.00%	1.00%	-	-
Preservative	3 ppm	3 ppm	3 ppm	3 ppm	-	-
Perfume	0.10%	0.10%	0.10%	0.10%	-	-
Sodium hydroxide	0.10%	0.10%	0.10%	0.10%	0.10%	0.10%
Water	Balance		Balance	Balance	Balance	Balance

_

	G	Н	ı	J	К	L	M
co-polymer	1.0%	-	-	-	-	1.0%	-
#7							
co-polymer	-	1.0%	-	-	-	-	-
#8							
co-polymer	-	-	2.0%	-	-	-	1.0%
#9					i		
co-polymer	-	-	-	2.0%	-	-	-
#10			i			2.504	
co-polymer	-	-	-	-	1.5%	0.5%	-
#11			'				0.5%
co-polymer	-	-	-	-	-	-	0.5%
#5					4.50/	1.5%	
Sodium	-	-	-	-	1.5%	1.5%	-
dodecyl						1	
sulphate						_	
Sorbitol	0.7%	0.7%	-	2 00/	-	_	
Propylene	-	-	3.0%	3.0%	-	_	
glycol						_	1.0%
Sodium	-	-	-	-			1.070
borate			2.0%	2.0%			_
Sodium	-	-	2.0%	2.070			
citrate	00.00/	20.00/				_	_
Isopropanol	30.0%	30.0%	_	_	20.0%	20.0%	-
Ethanol	-	_	0.5%	0.5%	20.070		-
Dye fixative	-	-	0.5%	0.570	1	I	1

Polyethylen	-	-	-	-	-	-	2.0%
eimine Carezyme	-	-	0.1%	0.1% 0.3%	- 0.1%	- 0.1%	- 0.4%
Perfume Potassium	0.15% 0.1%	0.15% 0.1%	0.3% 0.1%	0.3%	0.1%	0.1%	0.1%
hydroxide Water	Balance	Balance	Balance	Balance	Balance	Balance	Balance

	N	0
Co-polymer #12	0.5%	-
Co-polymer #13	-	0.75%
water-solubilising	0.50%	-
agent 1		
water-solubilising	-	0.75%
agent 2		
water-solubilising	0.25%	0.25%
agent 4		
Diethyleneglycol	0.75%	0.75%
Cyclodextrin	0.5%	0.5%
Preservative	3 ppm	3 ppm
Perfume	0.10%	0.10%
Sodium hydroxide	0.05%	0.05%
Water	Balance	Balance